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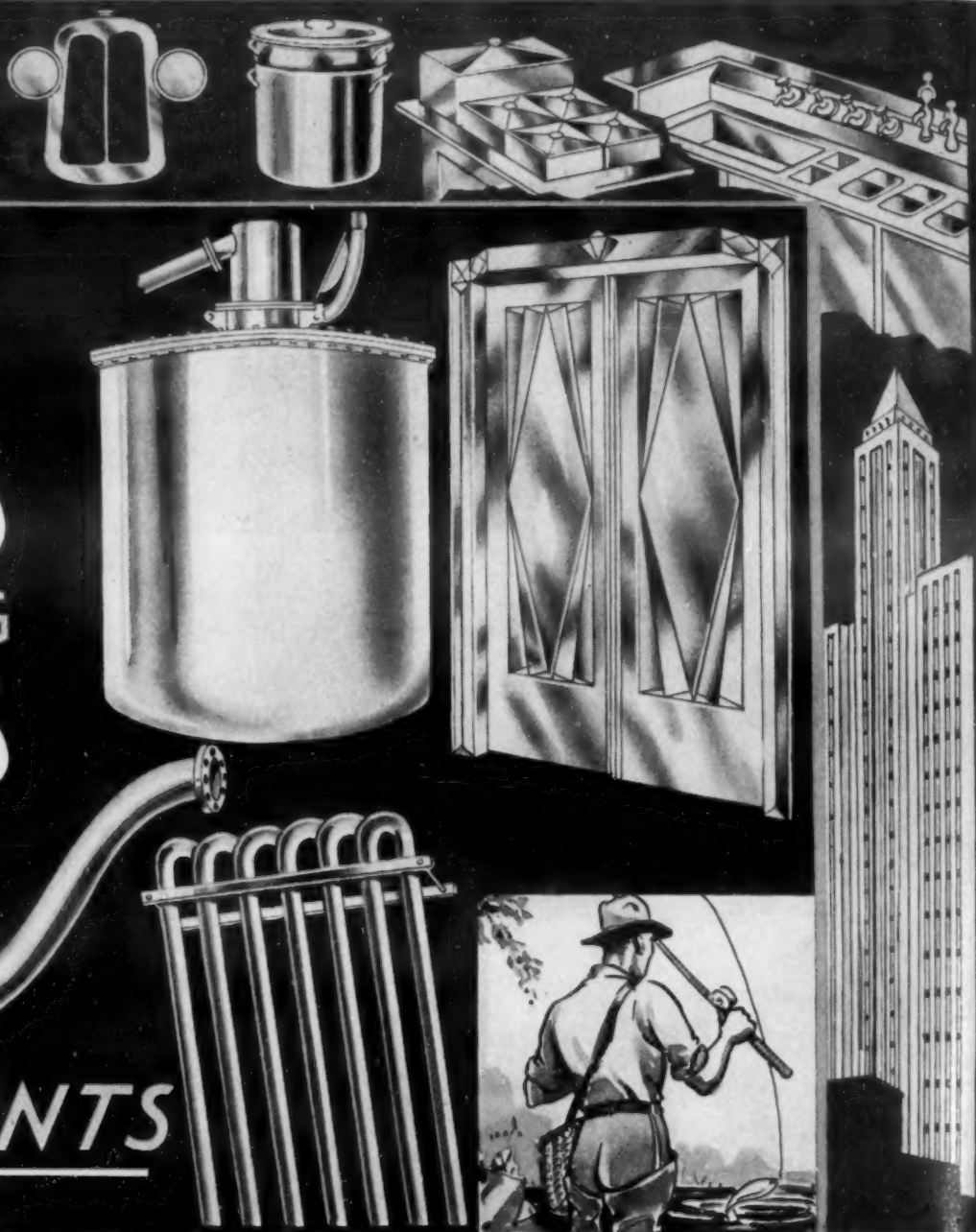
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EDITORIAL COMMENT

The Employment Exchange

NOW that all trustworthy "index" figures indicate that business has already begun a definite and increasing upward swing, METALS & ALLOYS announces the establishment of what for want of a better name we simply call an "employment exchange." Readers can register with us, giving information as to their training and experience, kinds of jobs which will be considered by them as well as salary expected. Openings which come to our attention will be referred to them immediately.

The question suggests itself: Why was not this step taken a year ago? A year ago we would only have been duplicating the work of engineering society committees and other organizations maintaining the usual form of employment exchange; a year ago, even with the best intentions, we could have done nothing to *create* opportunities.

Jobs for engineers in metal working and producing plants are not plentiful even at the present moment and the immediate future is the concern of every unemployed engineer. Some of the large companies have held their technical staffs on full time. Those companies forced to reduce their engineering force or to cut it off entirely will now slowly build it back as times continue to improve. These latter companies will be in the market for men. Any readers who will advise us of such openings will be performing a service to both parties concerned.

Our interest, though, is mainly in those companies who have never felt the need for technical control. Modern plant operation calls for precision methods of production and inspection. Standards have risen above those of 1929, while selling prices dropped again and again. At this time employers are seeking ways to economize, ways to make and increase profits. It is difficult to imagine an employer, who never has had the advantage of technical advice, buying a "pig in a poke," which is the thing most of the unemployed expect of him. At the same time he would be foolish indeed if he overlooked an opportunity to build his business or save it from possible ruin.

With the steady improvement in products, even the smaller manufacturers must institute technical control. This provides an unusual opportunity for unemployed metallurgists to sell the idea of technical control to the metal working and metal producing plants.

Too many see opportunities afar by craning their necks over those at home. If the unemployed engineer will study the problems of someone known to him, or of someone to whom he can be properly introduced, he will find that the company or companies have problems even if the plant is in his home town.

In many cities five or ten plants manufacturing non-competitive products are located. If one of these com-

panies cannot employ the full time of a man it may be possible that they will consider a part-time arrangement. It may even be possible that some of these plants previously employing the full time of a metallurgist cannot do so now or for some time to come. These companies will also be prospects for a part-time arrangement.

It should be possible for the unemployed engineer to prove his worth to several of these plants and sell them the idea of buying part of his time on a monthly retainer basis, with each of five companies paying him a monthly retainer fee. If there be need for analytical work and testing or research work for which the required equipment is not available, this could be sent out to university or consulting laboratories. If equipment is available at one plant, but the usual operator was let out, that plant could be made headquarters and that firm's share covered or partly covered by use of equipment.

Probably few who read this will be in need of employment, but all number among their friends and acquaintances some who are. Let everyone help to push such a campaign, for by doing so many good purposes will be served. The jobless metallurgical engineers may be employed, industries may be helped and the possible future usefulness of metallurgical engineering materially broadened by the introduction, at this critical time, of metallurgical engineers into new companies and industries.—RICHARD RIMBACH



New Member of METALS & ALLOYS Editorial Advisory Board

We are glad to announce that Dr. R. F. Mehl, Director of the Bureau of Metallurgical Research, Carnegie Institute of Technology is now a member of our Editorial Advisory Board.



New Officers of A. S. T. M.

The recent election of officers of the A.S.T.M. resulted in the election of Mr. Cloyd M. Chapman as President, Mr. William H. Bassett as Vice-President and the following as members of the Executive Committee: Messrs. H. A. Gardner, J. O. Leech, J. T. MacKenzie, J. C. Ramage and H. S. Vassar. Mr. Bassett and Dr. MacKenzie are members of *Metals & Alloys* Editorial Advisory Board.



Charles H. Marshall, until recently a member of the Technical Staff of The Bell Telephone Laboratory, Inc., has joined the staff of Lucius Pitkin, Inc., Consulting Chemists & Metallurgists as Materials Engineer. Prior to his service at the Bell Laboratories, Mr. Marshall had been in charge of the Physical & Mechanical Laboratories of The Westinghouse Electric & Manufacturing Company.

Introduction

COPPER has been used for centuries in building construction. One of the principal earlier uses was for the covering of the domes of cathedrals and monumental structures in all parts of the world in the Orient as well as the Occident. The green coloration or patina (verdigris) that develops through natural weathering on the exposed surfaces of copper was greatly admired by the architects of these early structures and tradition tells of various unusual methods used to accelerate its formation. The Egyptians and ancient Greeks also appreciated the beauty of natural patina and gave much thought to its formation. Bannister and Gowland in their book on ancient Egyptian Metallurgy quote a learned discussion from Plutarch's *Morals* regarding the subtle quality of the air at Delphi and the advantages of the use of olive oil in forming verdigris on bronze objects.

Appreciation of the beauty of this natural patina has persisted through the years and with the modern demand for color in building construction it is not surprising that architects have turned an envious eye toward the pleasing green offered by the patina on copper and copper alloys. The only objection to its greater use in modern architectural design has

been the time that is required for the patina to form by natural weathering, being, even in our polluted industrial atmospheres, of the order of 10 to 14 years.

The Copper and Brass Research Association, at the urgent suggestion of architects, undertook early in 1928 an investigation to develop methods whereby nature's processes might be hastened and the patina developed at

a rate more in keeping with modern production methods. It is the purpose of this article to present the results of this investigation.

Preliminary Studies

A review of the literature on methods of coloring copper showed it to be filled with innumerable formulae, some of wondrous complexity, by which the desired results could be obtained. A large number of the more reasonable solutions were selected for extensive trials. All of them gave a certain degree of coloring, some gave very pleasing shades, but none proved quite reliable.

Chemical Composition of Patina

The patina is traditionally referred to as being a basic copper

carbonate. U. R. Evans¹ in his book on Corrosion of Metals stated that "In the ordinary air of towns, the greenish gray deposit which appears on copper or bronze is mainly a basic carbonate (verdigris)" but he pointed out that Rathgen² and Fox³ had found chlorides to be present and

¹U. R. Evans. Corrosion of Metals. 2nd edition. E. Arnold, London, 1926, page 151.

²F. Rathgen. *Dingler's Polytechnisches Journal*, Vol. 301, 1896, page 44.

³J. J. Fox. *Transactions Faraday Society*, Vol. 19, 1924, page 913.

TABLE I.

Source and Description of Sample	Type of Atmosphere	Composition of Patina ^a						Composition of the Copper					
		Copper ^b %	Sulphate SO ₄ %	Chloride Cl %	Carbonate CO ₃ %	Sulphide %	Other Elements	Basic Copper Sulphate Cu SO ₄ · 3Cu(OH) ₂	Copper + Silver	Oxygen	Sulphur	Iron	Other Elements
Providence, R. I., sample of flashing and gutter from freight shed showing fine light green patina. Roof laid in 1850. Sample taken in 1928. Period of exposure about 78 years.	Industrial seacoast							Very large amount	99.504	0.1990	0.0025	As. 0.0942 Sb. 0.0088 Pb. 0.0084 Sn. 0.0064
Sample from railroad station. Period of exposure about 30 years.		49.70 (53.50)	20.2	0.00	0.00	0.00	Iron oxide silica, etc. 4.86	95.14	99.925				
New Haven, Conn., Woolsey Hall, Yale University. Roof laid about 1901 or 1902. In place about 25 years.	Industrial seacoast	49.94 (50.50)	19.06	Very small	Very small	89.1	99.915 copper 0.0241 silver	0.0526	0.0024	
Waterbury, Conn., Waterbury Brass Goods Corp. Sample of roof in place about 16 years.	Industrial 25 miles from seacoast	51.33 (51.96)	19.61	Very small	Very small	91.6	99.925				
Roofing from house at corner of Grand and State Streets. Light green uniform patina. Probably about 40 years old.		50.17 (52.60)	19.85	0.00	0.00	Small amount	Iron 0.84	90.0	99.841	0.130	0.0018	0.0016	
Prospect, Conn. (about 6 miles from Waterbury, Conn.) Copper telegraph wire. Light green patina. Length of exposure unknown.	Semi-rural		Very large amount	Very small amount	0.00	Very small amount	Cuprous oxide Large amount	Very large amount	99.961				

^aThe marked similarity of the composition of the patinas to the copper ore brochantite was noted at the time of analysis by the chemist, Mr. J. A. Crane, who had had recent experience with these ores at the Chuquibambata mines in Chile, South America.

^bFigures in () are theoretical copper values required to satisfy the formula Cu SO₄ · 3Cu(OH)₂.

^cThere was insufficient sample to make complete quantitative analysis. Qualitative tests showed corrosion products to be principally a basic sulphate of copper.

that Vernon⁴ had found a blue deposit on copper exposed to the open air in London to consist of sulphate.

In the fall of 1928 samples of copper roofing having a well developed patina were removed from the railroad station at Providence, R. I. Shortly afterwards similar samples were taken from Woolsey Hall at Yale University at New Haven, Conn., and from a factory building and a residence at Waterbury, Conn. The history of these roofs, their condition and results of chemical analyses of the patinas are given in Table I.

The results of the analyses were surprising in showing that in all instances the patina was a basic sulphate rather than the traditional basic carbonate. This was ascribed at the time to the probable influence of the sulphur dioxide contamination of present day industrial atmospheres.

The astonishingly false conception that had existed regarding the chemical nature of patina that forms on the surface of copper exposed to present day atmospheres was first publicly announced by Vernon and Whitby in their classical study⁵ of the surface patina on copper. In their studies it was found that the patina on the copper roofs of some of the oldest structures in England was also basic sulphate. The publication of these independent studies, made apparently at about the same time as the studies reported in the present work, were convincing proof that all reported methods of reproducing natural patina by accelerated means were based undoubtedly on a false premise.

Evans in a discussion of Vernon and Whitby's paper ascribed this false conception to the fact that most of the older data concerned products (patina) taken from copper or bronze antiques found in such places as the Egyptian desert, where the chief anions are Cl and CO₃. Thus it is quite natural that carbonates and chlorides should be found in such corrosion products.

In view of the data given in Table I the search for an accelerated method was of course directed toward methods of producing basic sulphate.

Synthetic Atmospheres and Simulated Atmospheric Attack

The possibility of accelerating patina formation by simulated atmospheric attack using higher concentrations of the corroding agents than normally present in the atmosphere suggested itself. An extensive study was made of the action of copper in various synthetic atmospheres of known carbon dioxide, sulphur dioxide, oxygen, air and moisture contents under various conditions of concentration, temperature and pressure. Since the

primary object of the studies was the commercial development of a method of producing a "natural" patina in a reasonable period of time the periods of exposure were confined to much shorter lengths of time than a more fundamental study of the probable mechanism of formation of the "natural" basic sulphate patinas would warrant, although of considerable scientific interest.

The studies showed definitely that basic sulphate patinas could be caused to form on the surface of copper when exposed to an atmosphere of sulphur dioxide in the presence of moisture but sufficiently consistent results could not be obtained within reasonable periods of time. The tendency in general was for copper sulphate to form on the surface of the copper. This was soluble and loosely adherent. Methods were developed for converting the sulphate to the basic sulphate but after such treatment the basic sulphate so formed lacked suitable adherence to the copper surface. Carbon dioxide appeared to have little influence either alone or in the presence of sulphur

dioxide which fact is in agreement with similar conclusions by Vernon and Whitby.

In a more recent report by Vernon⁶ of his extensive studies of corrosion of copper in synthetic atmospheres he has shown the intimate and critical relations that exist between the formation of basic sulphate on copper and the concentration of sulphur dioxide and relative humidity. His data show that for concentrations of sulphur dioxide below a critical value of about 1% the copper sulphate in the corrosion product is associated with excess base while with atmospheres containing one or more percent of sulphur dioxide the product is associated with excess acid.

This critical relation is probably a limiting factor in producing a basic sulphate patina on copper in synthetic atmospheres within commercially practical periods of time although tests indicate that the rate of reaction might be appreciably accelerated by working at higher temperature and pressures.

Incidentally it is of interest to call attention to this critical relation in atmospheric corrosion and the many futile attempts that have been made to develop accelerated laboratory methods of estimating relative resistance to normal atmospheric corrosion. In fact the great difficulty encountered in reproducing a patina on a copper surface, which is a natural corrosion phenomenon, is in itself an excellent illustration of the difficulty of developing representative accelerated test methods.

⁴W. H. J. Vernon. *Transactions Faraday Society*, Vol. 19, 1924, pages 875, 876.

⁵W. H. J. Vernon & L. Whitby. The Open Air Corrosion of Copper—A Chemical Study of the Surface Patina. *Journal Institute of Metals*, Vol. 42, 1929, page 181.

⁶W. H. J. Vernon. A Laboratory Study of the Atmospheric Corrosion of Metals—Part I. The Corrosion of Copper in Certain Synthetic Atmospheres with Particular Reference to the Influence of Sulphur Dioxide in Air of Various Relative Humidities. *Transactions Faraday Society*, Vol. 27, 1931, page 255.



Electrolytic Methods

The basic reactions of patina formation on copper in synthetic atmospheres involves the action of the gases oxygen and sulphur dioxide in the presence of moisture. These 2 gases together with water go to make up part of the molecule of basic copper sulphate. Consideration of the structure of the molecule of basic copper sulphate suggested that it might be possible to build it up in steps instead of expecting the whole reaction to proceed contemporaneously. To effect this the reaction between a solution of copper sulphate and solid copper hydroxide or hydrated oxide was employed. This consists of a three phase system in which the copper hydroxide is converted over to a more stable form when it is associated with copper sulphate in the molecule basic copper sulphate. This might be represented as: Cu SO_4 (dis.) + 3Cu(OH)_2 (solid) = $\text{Cu SO}_4 \cdot 3\text{Cu(OH)}_2$ m H_2O (solid). The importance, in order to obtain adherence, of effecting the formation of the green coating by acting on the molecules of copper on the surface of the sheet itself had been previously observed. The above reaction then would require the conversion of the surface of the sheet to copper hydroxide or hydrated oxide. It is conceivable that this might be accomplished in several ways. In short the method employed in this work was to bring about the oxidation of the copper by making it the anode in a suitable electrolyte. A bath of the following composition was found to be the most suitable:

- 2% sodium sulphate (anhydrous)
- .4% sodium hydroxide
- .1% sodium cyanide

At room temperature and at a current density of 30 m.a./dm.² the surface of the copper anode was covered with a black deposit. Upon removal from the bath this was found to be loose and spongy but comparatively thick. At a higher temperature (85°C.) the character of the coating changed. It was still quite black but extremely dense and adherent.

It was found that in accordance with the equilibrium given above both of these products could be converted to a light green product simply by heating at 95°C. for 1/2 hour in a 5% solution of Cu SO_4 . The adherence of the product was comparable to that of the oxide coating from which it was made. Unfortunately, while the desired reaction or effect

was obtained, neither product was practical. The coating produced from oxide formed at room temperature was of a very dense color but the material was very loosely adherent. The oxide coating produced at higher temperatures was very adherent but of such a small amount per unit surface that the coloring effect was unsatisfactory. Intermediate temperatures were investigated but the results were equally unsatisfactory.

Further development of the above method was postponed in view of the more promising results obtained by the ammonium sulphate process described below.

The Ammonium Sulphate Process*

In the numerous formulae given in the literature for coloring copper, ammonium chloride is often given as one of the essential constituents together with nitrates, carbonates and sulphates of copper and other compounds. It was somewhat surprising to note that ammonium sulphate had apparently not been considered. In view of the discovery that natural patina was a basic sulphate rather than the traditional carbonate and chloride, a study of the action of ammonium sulphate on copper was made.

It was found that copper when exposed to a spray of ammonium sulphate solution for about 24 hours acquired a rather dark green adherent patina similar in appearance to a natural patina although appreciably darker in shade. It was also found that when copper was subjected to alternate immersion in ammonium sulphate solution, as in conducting an alternate immersion corrosion test, a similar coating was formed. The coating (patina) was not formed, however, if the copper were continuously immersed in ammonium sulphate solution. This indicated that the formation of the patina was intimately associated with an oxidizing action of the atmosphere combined with the corrosive attack by the ammonium sulphate solution.

Based on these discoveries a commercially practical method has been developed for producing on the surface of copper and some copper alloys an adherent patina

similar in all respects to that produced by natural weathering. The patina may be applied in about 24 hours, depending on size of sample, as compared to the 12 to 14 years' exposure to the weather required for the patina to form by natural weathering.

From various practical considerations an

*Patent applications have been filed covering this process.

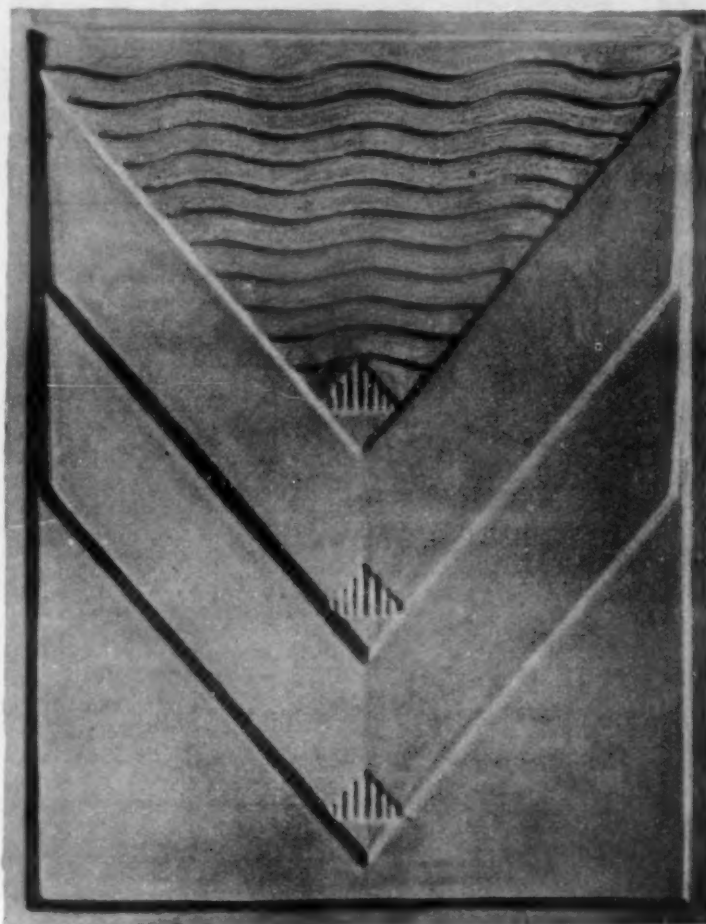


TABLE II. COMPOSITION OF SYNTHETIC PATINAS FORMED BY AMMONIUM SULPHATE PROCESS

	Copper Cu %	Hydroxyl Ion OH %	Sulphate SO ₄ %	Ammonium NH ₄ %	Water %	Basic Sulphate Cu SO ₄ · 3Cu(OH) ₂ %
Patina as formed by intermittent immersion treatment	52.37	21.00	21.28	0.90	4.45 Undetermined %	93.3
Patina after treatment in hot water	55.20	21.82	21.12	0.090	1.77	97.90
Pure Brochantite Cu SO ₄ · 3Cu(OH) ₂	56.16	22.63	21.21	—	— Undetermined %	100
Precipitate formed on walls and bottom of ammonium sulphate tank	54.50	21.84	21.60	.294	1.766	97.15

intermittent immersion system has been adopted for commercial application of the process although coloring may be obtained by any procedure which involves an intermittent wetting of the surface of the copper to be colored followed by exposure to oxidizing conditions which are most readily obtained by simple exposure to the atmosphere for a suitable period of time. This time interval between immersions in the ammonium sulphate solution is of paramount importance to the success of the process.

A 10% solution of ammonium sulphate has been found the most suitable. This is maintained at a temperature of about 60°C. Before suitable coloring is obtained the solution must be "conditioned." "Conditioning" occurs automatically by the running through of several batches of specimens. It may be accelerated by blowing air through clean copper chips immersed in the solution. As conditioning occurs the pH value of the solution increases, the solution takes on a bluish color and basic copper sulphate precipitates on the walls and bottom of the container. With continued use the solution tends to become less acid with accompanying decrease in quality of patina formed. This can be corrected by suitable addition of sulphuric acid. Best results are obtained with pH between 5.5 and 5.7.

The surface of the copper to be colored must be chemically clean in order to obtain good adherence. Cleaning is readily accomplished by the usual degreasing and pickling solutions such as are used in preparing surfaces for plating. It is essential that the surface be prevented from becoming tarnished during the final washing and transfer to the coloring rack, as tarnish will cause poor adherence.

The "primary tarnish" that forms on the copper surface during the ammonium sulphate treatment has been found to be of utmost importance. With specimens having considerable area, which require a longer oxidizing period between dips in the ammonium sulphate solution, it has been found desirable to first subject them to a repeated immersion treatment to develop a suitable "primary tarnish" uniformly distributed before starting the intermittent immersion cycle.

The color of the patina on completion of the intermittent immersion treatment is a dark blue-green, quite different in appearance from the natural patina. The composition of this patina is given in Table II. The dark shade is due probably to the presence of copper ammonium compounds. This patina is relatively permanent in industrial atmospheres but changes gradually on exposure to the lighter natural shade. In marine atmospheres the change is more rapid.

The change in shade is readily accelerated by immersing the specimen in hot water for about a half an hour after which the patina has a shade very similar to the

"natural" product.

An analysis of the final product is given in Table II.

The theoretical composition of pure brochantite is given for comparison, also the composition of the precipitate that must be present in the ammonium sulphate solution to obtain the desired coloring.

Comparison of the composition of the synthetic patina with natural patina (Table I) shows its very close identity.

It is of interest to note that Vernon has apparently worked out a coloring process also based on the use of ammonium sulphate solution. In a recent publication⁷ he makes the brief statement that "promising results have been obtained by treating the metal (copper) for about a week with a solution of ammonium sulphate followed by a short treatment with a solution containing basic copper sulphate in suspension."

Theory of Formation of Patina

The exact mechanism of formation of the basic sulphate in the ammonium sulphate process is not definitely known. The following theoretical considerations and hypothesis are suggested:

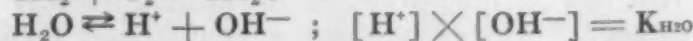
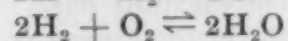
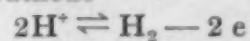
In the beginning we have a 10% solution of $(\text{NH}_4)_2$

SO_4 . Into this is dipped a sheet of copper which when thoroughly wet is withdrawn into the air. Immediately the solution begins to drain down from the sheet until the surface tension of the drip along the bottom edge of the sheet is able to support the film above. The resulting film remains on the sheet and is only diminished by evaporation. At once oxygen begins to diffuse into and through this film. It is quite reasonable to suppose that there will certainly be points on the surface of the copper where the oxygen concentration will have different values. This results in the setting up of so-called oxygen concentration cells. At a point of low oxygen supply the copper becomes the anode and corrosion takes place with copper ions going into solution. At a point of higher oxygen concentration the metal becomes cathodic. The initial effect of this is to make hydrogen ions discharge on the cathode with the production of a thin film of gaseous hydrogen. The oxygen present quickly destroys this film and the reaction continues. Because of this continual removal of hydrogen ions from solution there results a surplus of hydroxyl ions around the cathode. The equilibria thus set up are:

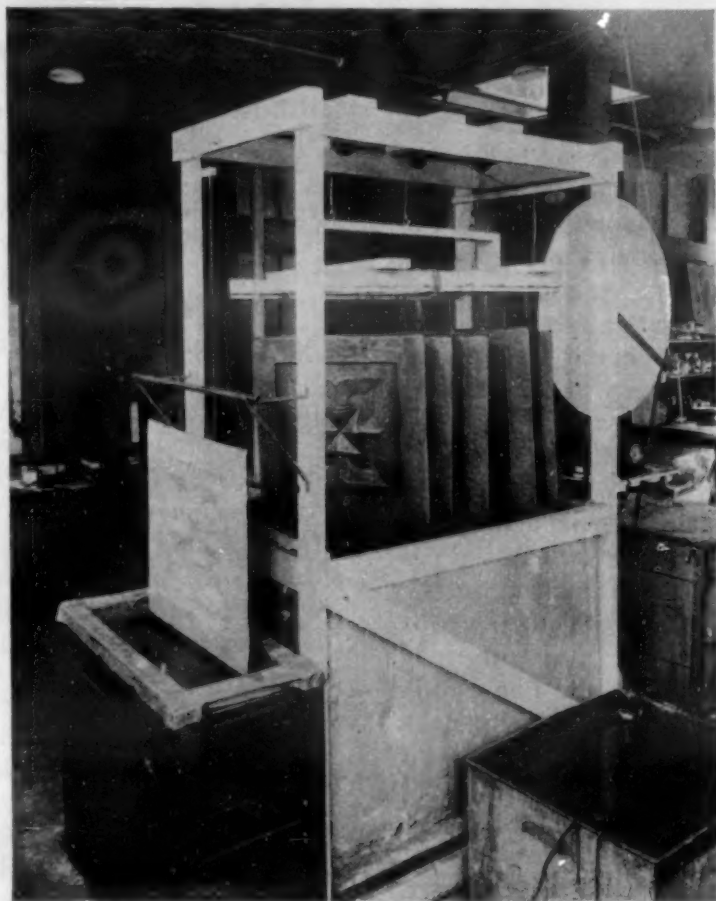
1. Anode



2. Cathode



⁷W. H. J. Vernon & L. A. Jordan, The Relation Between Corrosion and Paint, *Chemistry & Industry*, Vol. 50, Dec. 25, 1931, page 1050.



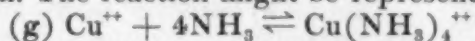
Laboratory Equipment Used for Coloring of Copper Spandrels by the Ammonium Sulphate Process Using the Intermittent Immersion Method

The equilibria existing in the solution initially are:

- (a) $(\text{NH}_4)_2\text{SO}_4 \rightleftharpoons 2\text{NH}_4^+ + \text{SO}_4^{2-}$
- (b) $\text{HOH} \rightleftharpoons \text{H}^+ + \text{OH}^-$
- (c) $\text{H}_2\text{SO}_4 \rightleftharpoons \text{H}^+ + \text{HSO}_4^- \rightleftharpoons \text{H}^+ + \text{SO}_4^{2-}$
- (d) $\text{NH}_4\text{OH} \rightleftharpoons \text{NH}_4^+ + \text{OH}^-$
- (e) $\text{NH}_4\text{OH} \rightleftharpoons \text{NH}_3 (\text{dis.}) + \text{H}_2\text{O}$
- (f) $\text{NH}_3 (\text{dis.}) \rightleftharpoons \text{NH}_3 (\text{gas})$

Reactions (a) and (b) are simply the ionization of a salt and water. Reactions (c) and (d) are secondary equilibria which are set up between the ions of the salt and water. These two reactions are associated with hydrolysis. The relative degrees of dissociation of the acid and the base determine whether the salt is acid or basic. In the case of ammonium sulphate the salt is acid (pH of 10% about 3.5). Of course the amounts of undissociated ammonium hydroxide and likewise of ammonia dissolved or as a gas are very small.

Both the anode and cathode reactions affect this equilibrium. Hydroxyl ions from the cathode and copper ions from the anode diffuse out into the layer of solution on the surface of the sheet. The cathode reaction does not add a new ion to the solution but reinforces the supply already present. This affects equation (b) and (d) directly. If (b) is displaced to the left hydrogen ion is removed and the pH of the solution is raised. As (d) is displaced to the left (e) and (f) are displaced to the right. This latter effect is manifested by the presence of a distinct smell of ammonia near the sheet. Also the pH is observed to rise as the dipping continues. The anode reaction however puts a new ion into solution, namely cupric ion. There are several possible reactions into which these ions may enter. As the new solution "ages" there is one reaction into which the appearance of the solution indicates that the copper ions must enter. This is the formation of the blue copper ammonium complex ion. The reaction might be represented as:



also the following relation holds where the quantities in brackets represent the concentrations* of the respective components. K_r is a constant dependent on temperature.

$$\frac{[\text{Cu}^{++}] [\text{NH}_3]^4}{[\text{Cu}(\text{NH}_3)_4^{++}]} = K_r$$

The next observation that is made is that the pH rises to a value slightly higher than and then comes back to 5.5 where it remains practically constant. The depth of blue of the solution does not increase appreciably from this point on, with continued treatment of the copper sheet. This shortly results in the formation on the walls of the tank or jar containing the solution of a light green precipitate. Analysis shows this material to have a composition practically identical with the natural patina product, i.e., $\text{Cu SO}_4 \cdot 3 \text{ Cu}(\text{OH})_2 \cdot \text{H}_2\text{O}$. Evidently then the solution has become saturated with this salt, which condition may be represented as follows:

(h) $\text{Cu SO}_4 \cdot 3 \text{ Cu}(\text{OH})_2 \cdot \text{H}_2\text{O} (\text{cryst.}) \rightleftharpoons \text{same salt (dissolved)}$

(j) dissolved salt \rightleftharpoons the ions Cu^{++} , OH^- , SO_4^{2-} . The concentration of the dissolved salt must be fixed since the solution is saturated, so therefore the product of the concentrations of the respective ions must equal the equilibrium constant or solubility product for this salt. This may be expressed as follows:

$$[\text{Cu}^{++}]^4 \cdot [\text{OH}]^6 \cdot [\text{SO}_4] = K_s$$

Evidently then the solution has so adjusted itself with respect to pH and Cu^{++} content that this relation holds. It is now possible to outline the method whereby a sheet of copper dipping into such a solution may become col-

ored with basic copper sulphate. Since the solution is saturated with basic copper sulphate as the sheet is withdrawn from the tank the equilibrium involving K_s must be satisfied. The anode and cathode reactions occurring in the film at the surface of the sheet proceed to furnish Cu^{++} and OH^- to the solution. Of course these immediately exert their influence according to K_s with the result that basic copper sulphate is ultimately precipitated.

It is possible that an intermediate reaction may set in here due to the relative speeds with which Cu^{++} and OH^- diffuse. The OH^- being the faster ion the chance occurs for a localized increase in NH_3 content and thus $\text{Cu}(\text{NH}_3)_4^{++}$. This local situation is ultimately made to revert upon meeting the stream of Cu^{++} from the anode. The following simple experiment served to demonstrate this fact. To a quantity of "aged" solution a small quantity of NH_4OH was added with the resulting increase in blue color. An addition of saturated copper sulphate solution slowly removed the blue color and caused the precipitation of basic copper sulphate.

The anode and cathode reactions produce equivalent quantities of $\text{Cu}^{++} + \text{OH}^-$ whereas the basic copper sulphate removes 8 equivalents of Cu^{++} for 6 equivalents of OH^- . This on first thought should increase the pH but this is compensated for by the reactions (d), (e), (f) with the ultimate result that NH_3 is lost to the air and basic sulphate is precipitated onto the surface of the copper.

Summary

A process has been developed by means of which copper and certain copper alloys may be given an adherent green surface coloring (patina) which is practically identical in composition and appearance with the patina that forms on copper surfaces after a period of years when exposed to the atmosphere. The process makes available to architects copper in almost any desired architectural form having a surface patina similar in all respects to the natural patina.

The process must at present be applied to copper shapes before installation on the building. Types or spandrels that have been successfully colored are shown in the illustrations.

The synthetic patinas are believed to be permanently resistant to weathering. To date specimens exposed to a severe industrial atmosphere for a period of over 6 months during which time they were wet down by heavy "showers" 4 times daily have shown no change or lack of adherence. Similarly specimens exposed to a strictly marine atmosphere for five months, including severe winter weather, have shown no failure or tendency of the patina to break down.



Tin Supplied to the United States Almost Entirely by Importation.

The value of tin required by the United States is much greater than that of any other mineral product that is furnished almost entirely by importation, points out the United States Bureau of Mines, Department of Commerce. Tin imports have annually exceeded \$60,000,000 in value but domestic mine production was worth less than \$11,000 for the last year of record. Moreover, the net imports of tin exceed, in value, those of any other mineral product and the metal is among the ten leading commodities imported.

The principal tin-consuming industries are food-packing, automobile manufacture, and building. The packing of food is relatively stable from year to year but the automobile and building industries are subject to wide variation of tin consumption, says Charles White Merrill, in a recently issued Bureau of Mines report.

*Strictly speaking the quantities in brackets represent the "activities" or effective concentrations of the various groups in solution.

FATIGUE OF METALS

A Backward Glance

By H. F. MOORE*

JUST AS the financial affairs of a nation seem to go in cycles of alternate "boom" and depression, so some scientific questions seem to have alternating periods of renewed interest and of quiescence. This is true of the subject popularly, but inaccurately, known as the "fatigue of metals." Its first period of interest centers around the work of Wöhler between 1859 to 1870 and the work of Bauschinger immediately following. The main impulse which started this period of experimentation was due to the introduction of railroad rolling stock fitted with "live" axles. A "live" (turning) axle is a member particularly subject to fatigue failure in service, on account of the reversal of flexure it undergoes for each revolution. The principal form of fatigue testing machine of today dates from this period and is nothing but a laboratory adaptation of a rotating loaded car axle.

The experimental results of this early period gave some evidence of a limiting stress below which failure would not occur, under an indefinitely large number of cycles of stress, although from the modern viewpoint the evidence was not very conclusive. This limiting stress was supposed to coincide with the "true" elastic limit of material. It was before the days of the metallurgical microscope and the assumption that solid bodies might be regarded as homogeneous and continuous was rather uncritically accepted. The idea of a "true" elastic limit, although even at that day it had been criticized by some of the leading writers on materials, became a dogma, almost a superstition, of the materials engineer.

The idea was generally accepted that repeated stress caused a mysterious change in a metal. The metal "crystallized," and became brittle. It lost its "life" and toughness; it became "fatigued." These ideas have been abandoned, but the term "fatigue" still sticks, and there are still some, though fortunately their number is decreasing, who still speak of metal "crystallizing" under repeated stress.

Between the period of Wöhler and Bauschinger, and the revival of interest in the strength of metals under repeated stress, which began about 1910, several fairly extensive series of experiments were made, notably at Watertown Arsenal, and there appeared one almost uncanny prophetic anticipation of the modern view in Kipling's story "Bread Upon the Waters" published in his collection of short stories entitled "The Days Work."

The beginning of the second period of active interest in the failure of metals under repeated stress was ushered in by the work of Ewing, Rosenhain, and Humphrey, who used the metallurgical microscope to examine metals under repeated stress. At once the idea of "crystallization" was seen to be wrong. Metals were always crystalline, and under heavy repeated stress the crystals were broken up into thin laminae which slid over each other, and this sliding developed into cracking and spreading fragmentation. Moreover, the metallurgical microscope showed the essential inaccuracy of the assumption of homogeneity and continuity when applied

to solid metals. The metallurgists themselves did not fully realize the far reaching consequence of their work.

The first period of interest in fatigue of metals centered around German laboratories. The next period of interest centered, at least at its beginning, around English and American laboratories. Mention should be made of the work of Prof. Basquin at Northwestern who, in 1910, published a paper before the American Society for Testing Materials in which he challenged the idea of a limiting stress below which failure would not occur and suggested that a safer viewpoint, on the basis of then existing data, was to assume a relation which involved the variation of the period of endurance of a metal with some inverse power of the stress. His theory has not been substantiated by further experiments, but it was of very great use in stirring up thought, and in showing the need of further test data. It was a good bit of *engineering* reasoning, based on the principle that if we cannot be sure that a theory is true it may still be of some use if we are sure that it errs on the *safe* side.

The second "boom" period in fatigue of metals investigations is partly a product of the great war, and the history of investigations which have been carried out since that period, mainly in England and America, but lately in Germany, is too well known to need repetition. Out of these experiments has emerged the generally accepted fact that there is a limiting stress (called "endurance limit" or "fatigue limit") below which failure will not occur under at least hundreds of millions of repetitions, that the maximum stress which a metal will stand is a function of the range of stress to which it is subjected (although the form of this function is still rather uncertain), that the limiting stress under cycles of shear is about half as great as the fatigue limit under cycles of tensile and compressive stress.

On the experimental side, fatigue testing equipment has become common in a large number of laboratories. Fatigue tests have not yet appeared in standard specifications, largely on account of a long time necessary to carry them out. Many attempts have been made to discover a short-time test which should give us a reliable index of the endurance limit, but so far none have been discovered. The attempt to discover an *elastic* limit which would indicate fatigue strength has been entirely unsuccessful, and the tensile strength has been found to have a closer correlation with fatigue strength than any other physical property, although this correlation is not close enough to allow it to be used as an accurate quantitative index. The whole problem of the interrelation of chemical action and the strength properties of materials has been given an enormous impetus on the work of McAdam and Haigh on fatigue strength of metals subjected simultaneously to cycles of stress and the action of corrosive liquid. So mild a corrosive liquid as ordinary water has been found to reduce enormously the fatigue strength of a metal if it is applied *simultaneously* with cycles of stress. Means of protection against such

(Continued on Page 207)

*Research Professor of Engineering Materials, University of Illinois.

Importance of Fatigue of

By T. A. SOLBERG*

WITH THE increasing use of metals and alloys in industry and the increasing knowledge of their various properties and specific applications there comes also a gradual refinement in engineering design. Each new development, be it a new alloy of better physical properties, an improved method of heat treatment, new knowledge of age hardening or working, or in short, any process resulting in better materials, is reflected back to the designer who must constantly revise and improve his art to keep step with these upward trends.

Perhaps one of the most recently explored fields which is making felt its effect on general design is the behavior of metals under conditions of fatigue and "corrosion-fatigue." It is certain that the gradual accumulation of knowledge in this field has a tremendous influence on many phases of design. In referring to Dr. D. J. McAdams' work on corrosion fatigue, the effects of which he first noted in 1926 during experiments in fatigue, the following opinion has been expressed,¹ "McAdams' conclusions on corrosion fatigue affect and change the very foundation of engineering design of all structures in which corrosion fatigue can play a part."

Ordinary fatigue on the other hand has been noted by many investigators of which Wöhler's work between 1852 and 1871 was the first of note. Despite this early work little constructive experimental and research work on this problem was done prior to 1919. Since that time the knowledge of the phenomenon of fatigue of metals has increased rapidly and its effect on design and on the application of metals and alloys to specific uses has been far reaching.

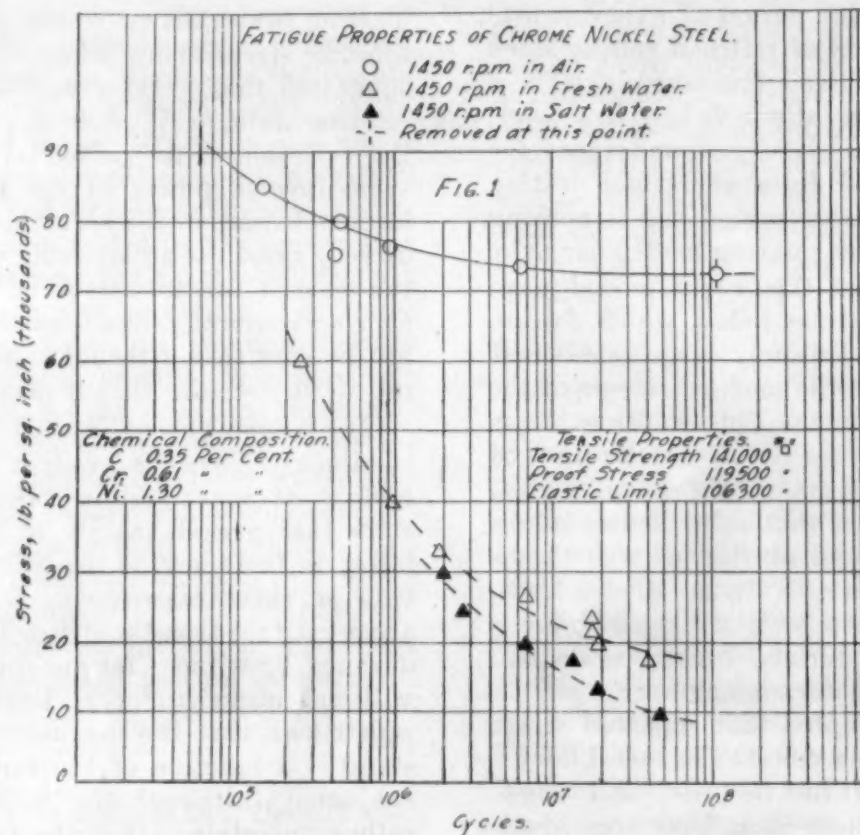
The use of the word fatigue in connection with certain metal failures has been confusing for the reason that the so called failures are not the result of any "tiredness" or "weariness" of the metal structure, which the word fatigue implies. In the past—and to some extent even now the term erroneously persists—the characteristic fatigue failures were ascribed to "crystallization" of the metal because of the crystalline appearance of a portion of the fracture. The application of the microscope, and more recently the X-ray, to metallography has proved conclusively that no crystallization or other change in the metal structure takes place when it is subjected to stresses that result in fatigue failure.

Briefly, a fatigue failure of a metal will occur when the metal is subjected to a sufficient number of applications of repeated stress which exceed a limit known as the endurance limit. If the stresses are below this limit an indefinitely large number of reversals of stress may be applied without failure. Fig. 1 shows graphically the determinations necessary to establish this limit and will help to explain much in relation to the whole phenomenon. For steels subjected to alternating tension and compression, the endurance limit is approximately 50% of the tensile strength of the material. In other words if the steel is subjected to cycles of repeated stress in tension-compression greater than 50% of its tensile strength, failure will occur after a definite number of cycles depending on the excess of the stress above the endurance limit. In alternating torsion, however, the endurance limit for steel varies between 20 and 30% of the tensile strength. No definite relationship appears

to exist between the endurance limit of a metal or alloy and the usual physical properties, other than the approximate relation with tensile strength or hardness of steel, within certain limits, and even this approximation fails to hold in non-ferrous materials.

Another term often used in connection with fatigue is the endurance range. In the usual rotating beam or cantilever tests the range through which the specimen is stressed is the spread between the maximum stress in tension and the theoretical equivalent stress in compression. The endurance range as determined by tension compression tests is therefore twice the value of the endurance limit determined from these tests. In

torsion endurance testing the specimen is stressed the same amount but in opposite directions and the endurance range is therefore twice the torsional endurance limit determined from these tests. McAdam conducted experiments in alternating torsion in which he varied the stress range, or spread of stresses, both in extent and in position, and as a result of this work found that for any location within the elastic range of the material the endurance range did not vary more than 10%. He also found that there is a definite point beyond which either limit of the endurance range could not be moved. This point is in the vicinity of the static elastic limit. This knowledge is of service in evaluating materials for purposes where alternating stresses are to be encountered, for it follows from the above that the one having the highest ratio of elastic limit to tensile strength is in-



herently the safest choice.

The mechanism of fatigue failure is a complex one and no complete satisfactory explanation of all the factors involved has been given. It is well known that at any sharp discontinuity of a stressed material there is a concentration of stress or an intensification of average nominal stress. The discontinuity may be at a small inclusion of foreign material, a sharp change in section, at an inadequate fillet, at an oil hole or screw head, at a keyway or even at a fine tool cut which may only be microscopic in size. If the stress concentration in such a region is sufficient to raise the nominal stress above the endurance limit of the material, failure of the immediate adjacent metal structure occurs. This becomes the origin of a progressive fatigue failure. When the available metal remaining is stressed beyond the ultimate strength, abrupt failure takes place and it is this final fracture which has the crystalline appearance so characteristic of fatigue failures. The area which represents the relatively slow progress of the crack usually appears smooth because of the rubbing action between the ruptured faces occasioned by stress reversal. Typical fatigue failures are described and illustrated at the end of this paper.

"Corrosion-fatigue," briefly, is a special case of fatigue in which the initial crack or point of stress concentration is found at an area which has become pitted or corroded by some agent, usually fresh or salt water. Many ramifications of the corrosion problem are to be had—corrosion simultaneous with fatigue, corrosion prior to fatigue, intermittent corrosion and fatigue, etc.

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**Lieutenant, U.S.N.

¹H. W. Gillett. Discussion, *Proceedings American Society for Testing Materials*, Vol. 27, Part 2, 1927, page 141.

Metals to Engineering Design

AND H. E. HAVEN * *

Dr. McAdam discovered in 1926, when testing fatigue specimens, that a stream of fresh water lowered the fatigue limit radically in the case of steel. With salt water the reduction was even greater. After much experimental work, these new fatigue limits McAdam denoted as "corrosion-fatigue" limits.

In regard to the combined effect of corrosion and stress as found in steam boilers, special conditions which may exist have given rise to the term "caustic embrittlement" or embrittlement of boiler plate. Definite examples of boiler failures have occurred in the Naval Service which can be ascribed quite definitely to the effect of corrosion and fatigue. On the other hand in the industrial field there have been numerous examples of failures in boilers which have operated under conditions which apparently fulfill those under which embrittlement of boiler plate may occur. Caustic embrittlement, or cracking of boiler plate, occurs generally in places of high stress concentration such as seams, between rivet holes, in the rivets themselves, in flanges or straps. In these locations, if the boiler water is relatively high in alkalinity, high concentrations of caustic may accumulate as the result of leakage. It is believed, that although caustic embrittlement or cracking of boiler plate may be a more complex phenomenon than that explainable by corrosion fatigue, that many of the failures described as caustic embrittlement failures are in reality special cases of corrosion with stress in which the corroding agent is the high concentration of caustic.

In order to shed more light on these subjects, however, it will be necessary to conduct more elaborate and complex experiments than those necessary to determine corrosion fatigue limits. The variables, frequency, stress and corrosion conditions, as well as temperature and pressure effects, must be evaluated. The experiments must also be run in two stages, namely corrosion and fatigue stages, since corrosion or other chemical attack is the fundamental problem involved.

The damaging effect of corrosion with or without stress is a factor which should not be overlooked in the design of any machinery part or structure. But, as previously stated, the designer cannot adopt an ordinary corrosion fatigue limit as direct basis for his design calculations. This is because in the ordinary corrosion fatigue testing no attempt is made to evaluate the proportion of net damage caused by fatigue and that resulting from corrosion unless specially conducted experiments involving all the conditions to be met are carried out. The usual corrosion-fatigue limits can be used only as a guide, and as an aid in determining the relative value of materials.

From a consideration of the above it can be seen that the constant acquisition of information regarding the fatigue and corrosion fatigue properties of metals and alloys should result in a gradually growing refinement of the designer's art. The well known and much abused factor of safety so commonly used finds considerable enlightenment in these phenomena.

Many of the common causes of failures were formerly associated with and considered as good design until the revelations of fatigue testing demonstrated that radical changes were necessary. An ordinary fillet is no longer tolerated. It must be proportioned in accordance with the stresses encountered so that failure cannot occur in this region. The failure of a piece of metal anywhere in a filleted arch frequently bespeaks inaccurate design. For important purposes, bolts, tie rods and

other members on which screw threads are cut are now proportioned so that if failure ever occurs from any fatigue factor, the location will not be at the base of a screw thread. The same rule holds wherever a change in section or a discontinuity of section occurs. If an oil hole must be provided in a shaft it must be placed so that there will not occur in its wake any stresses which, because of the possible concentration normally produced by the hole, would contribute to fatigue failure. These are only a few examples where knowledge of fatigue phenomena has led to improvement in design. And closely related to the design of machines and structures is the proper determination of the uses of metals and their alloys for the specific applications for which they are best suited. Knowledge of fatigue and corrosion-fatigue properties serves best to specify the optimum material for any specific purpose.

In airplane construction, as an example, from wing tip to wing tip and from rudder to engine, economy of weight consistent with maximum safety is absolutely necessary. Experiments for determining the alternating torsional endurance and corrosion fatigue properties of airplane wires are now in progress and yielding much valuable information. This is only one example of work being done in this field to promote increased safety in construction.

It was in the metallurgical laboratory of the U. S. Naval Engineering Experiment Station, Annapolis, Maryland, where Dr. McAdam conducted, first his important endurance testing experiments, and later in 1926 encountered and studied corrosion fatigue phenomena. This laboratory since that time has investigated many service examples of both types of failure. One function of this Station is to investigate and determine the causes of failures of all classes of materials used in the field of Naval engineering.

A short description will be made, and history given of a few of the more important failures with photographs to illustrate the appearance of the failure. In some instances rupture had not occurred when the defect was discovered.

Case A.—Failed crank shaft of service airplane engine. Characteristic appearance shown in Fig. A. The chemical analysis, physical and metallographic examination showed the material of this crankpin to be of normal quality. The failure of this crankpin was due to fatigue. That is, the pin was subjected to repeated stresses which were higher than the endurance limit of the steel. A crack started at the crankpin oil hole and progressed in both directions. Discovery of an incipient fatigue crack at the edge of the oil hole

90° from the helical fracture—Fig. A—makes it appear likely that the member was subjected to alternating torsional stresses incident to critical vibration. An accurately formed and smooth finished hole is in itself, a cause of stress concentration. The imperfection in the form and finish of the oil hole in this crankpin further intensified this condition. In addition to these factors it is likely that the engine was operated at or over its critical speed.

Case B.—Failure of a submarine propelling motor shaft. This failure was the result of building up by electric welding of the shaft surface to restore the worn and pitted area of the journal to its original dimensions. Failure resulted because it was not appreciated that the turning down of the shaft prior to welding produced a sharp change in section which resulted in stress concentration in this region, despite the fact that subsequent welding tended to correct the condition. The welding also stopped in a filleted portion of the shaft. In such a case the shaft should have been turned so that a long fillet was made where the turned section stopped. Fig. B shows the rather sharp fillet as well as poor welding caused by the wire

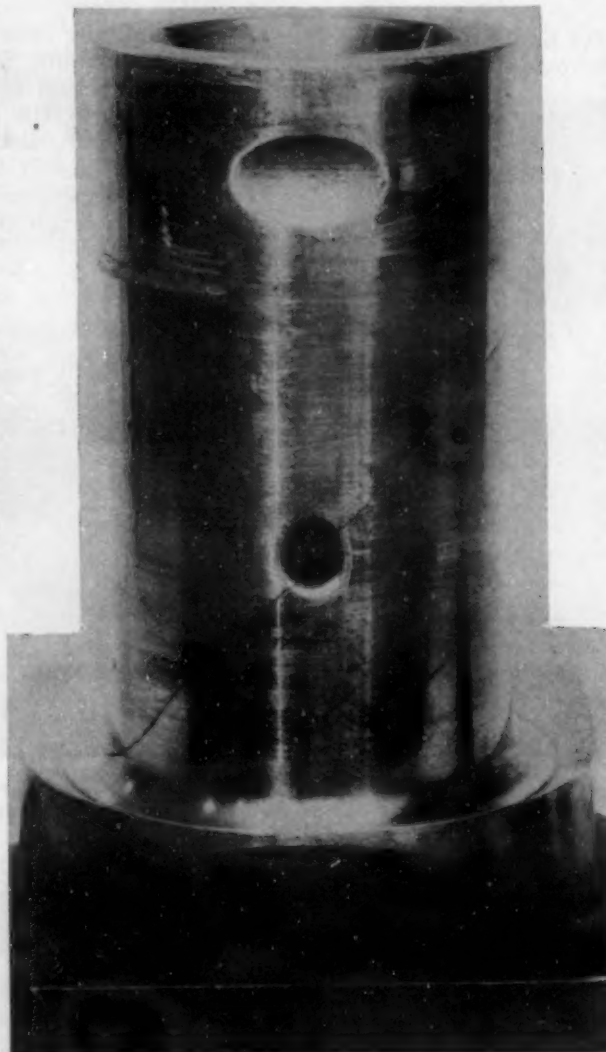


Fig. A. Fatigue failure of airplane engine crankshaft. Crack started at oil hole.



Fig. B. Submarine propelling motor shaft, 8 1/4" diam. Figure shows portion built up by welding to restore original dimensions. Failed in plane at end of welding which was also in a filleted area of the shaft.

of bare electrodes. With a large radius fillet and proper welding this repair might have restored the shaft to approximately its original condition. The figure shows the welded portion of the shaft which ends at the fracture.

Case C.—Failure of a large stern tube shaft. In this case the ultimate rupture had not occurred but was impending at any moment. The shaft was of hollow construction (about 14" X 9"), covered with a shrunk-on composition sleeve. During docking and overhaul a slight seepage of rusty water at a point which proved to be a joint between adjacent sleeve sections led to further examination. Sea water had gained access to a small area of the steel shaft and a corrosion-fatigue crack

was well underway with failure imminent. Fig. C1 shows the corroded appearance of the shaft surface and the start of the crack is also visible. For test purposes the shaft was carefully sectioned in such a way as to develop or follow the contour of the crack. Fig. C2 shows a portion of the section of the shaft after this work was completed. All of the area shown in the figure represents the boundary of the corrosion-fatigue crack. This is considered an excellent example and the manner in which the crack was finally developed has given interesting and valuable information. This case illustrates the importance of considering the effects of corrosion and fatigue in the design of members which are subject to these conditions. In this instance, protection from corrosion was afforded to this portion of the shaft by the metal sleeves. Slight access of water to a small area of the shaft was probably the direct result of vibrations which existed at various times when the strut and stern tube bearing clearances were at or near the allowed tolerances. This vibration no doubt caused leakage at the sleeve joint



Fig. C-1. Hollow turbine shaft of direct drive vessel showing corroded surface and crack.

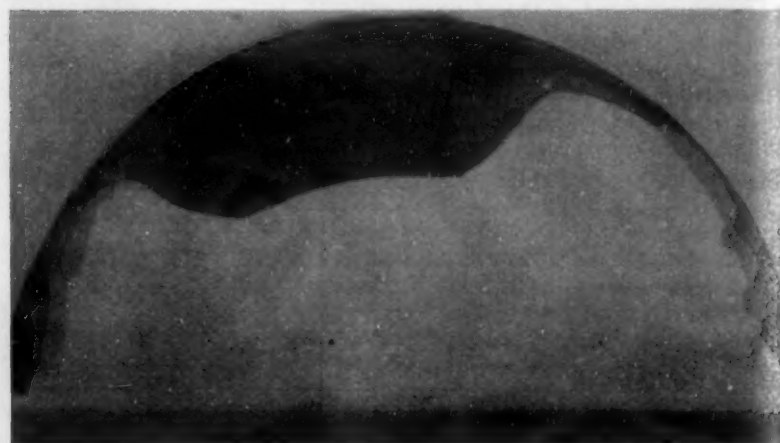
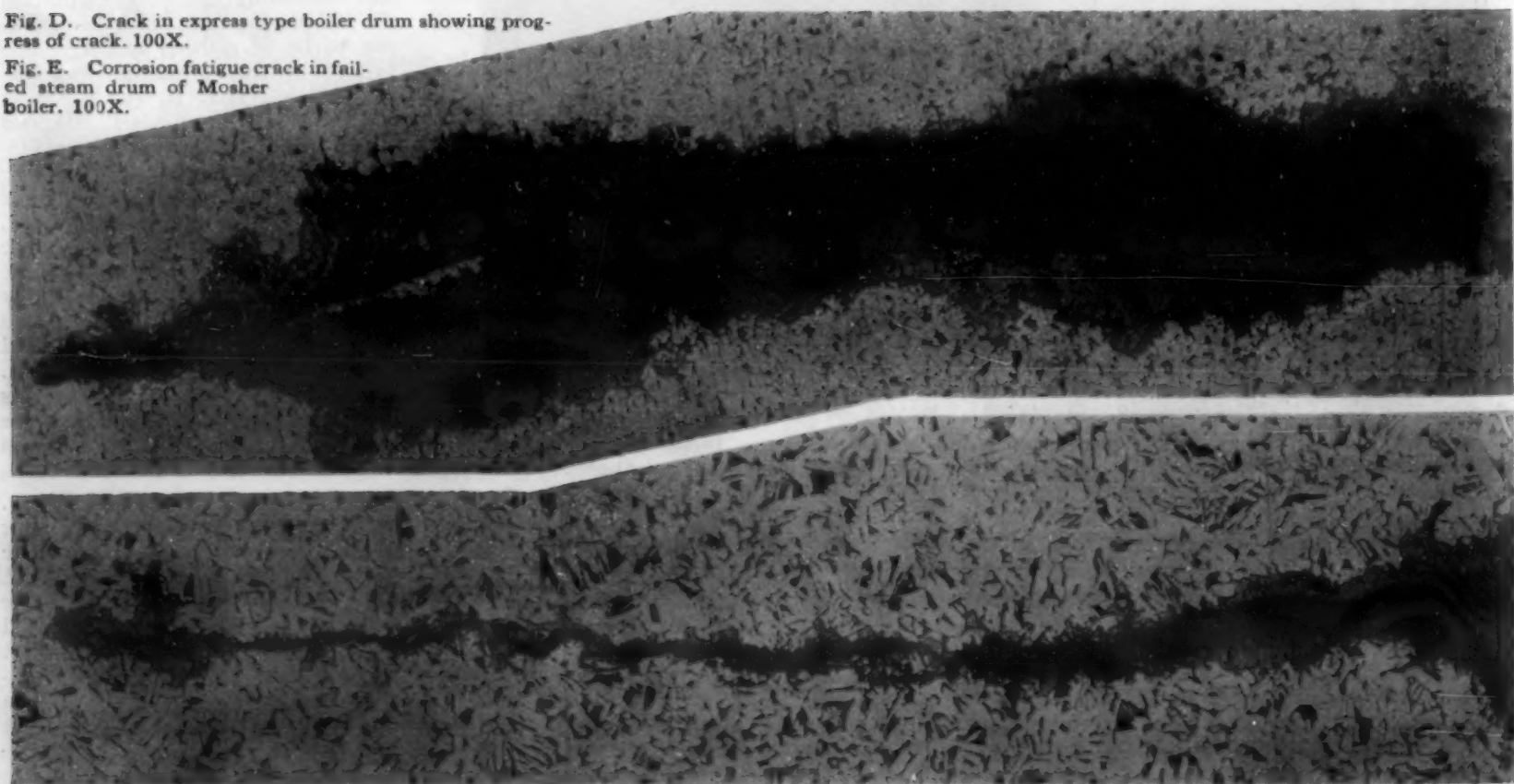


Fig. C-2. Half section of stern tube shaft shown in Fig. C-1, showing progress of crack. Rupture was imminent.

Fig. D. Crack in express type boiler drum showing progress of crack. 100X.

Fig. E. Corrosion fatigue crack in failed steam drum of Mosher boiler. 100X.



which was located at such a point with relation to the bearings, that maximum stresses under vibratory conditions occurred in this region. Corrosion and fatigue together, then had full play.

Case D.—Impending failure in "A" type Boiler Mud Drum. This was another case where failure had not yet occurred. The location of the crack showed that it originated at a change in section where the computed factor of safety was not only below accepted standards, but in addition, had been further lowered by the reaming of the holes in the drum in order to assist in rolling in the tubes. As the boiler had never had alkalinities in excess of $0.5\% \times a$ 1 normal solution (based on sodium carbonate) the action cannot properly be ascribed to caustic embrittlement. The photograph (Fig. D) was taken through the crack and shows clearly its progress and the combined mechanism of corrosion simultaneous with stress which was leading rapidly towards failure.

Case E.—Mosher Boiler. Fig. E shows a photomicrograph of a corrosion-fatigue crack which developed in the steam drum of the Mosher Boiler formerly installed at this Station. This boiler exploded in August 1926 and the cause was without doubt due to corrosion-fatigue. At the top of the photograph the corrosion products are very prominent while in the lower portion these products are very small in proportion. Evidently the progress of the failure can be divided into two stages. In the first stage the crack developed and increased in size mainly as a result of corrosion while fatigue entered into its increase a comparatively small amount. In the second stage, the structure having been weakened, fatigue began to have a more pronounced effect and the crack began to extend with a relatively high speed. In this stage the amount of corrosion per cycle was considerably less than in the first stage and the fatigue factor was of more importance than the corrosion factor.

Case F.—Main Drive Reduction Gear Pinion. The condition of this pinion was the result of fatigue occasioned by compressive stresses above the fatigue limit of the steel. Under these conditions small particles of material were sheared from the surfaces of the teeth resulting in the pits shown in Fig. F. The actual load transmitted by this gear was evidently in excess of the compressive endurance limit of the material. In the case of a gear pinion a complete cycle of stress between zero and a maximum and return to zero occurs with every revolution. This condition developed after a relatively short period of service. The metallographic and physical properties of the material were fairly normal with somewhat more than the non-metallic inclusions desirable.

Case G.—Airplane engine crankshaft extensions. Figs. G1 and G2 show failures of the same part of two different engines. This extension is a connecting shaft between the inertia starter and the gears turning over the crankshaft when starting the engine. The part is of course subjected to a very high suddenly applied load when the starter clutch is engaged. Failure of both of these parts was caused because the upper limit of the range of stress of these applied loads was above the endurance limit of the material. The tensile, ductile and elastic properties of the material in these pieces are quite normal for steel of this composition when quenched and drawn at a relatively low temperature. It will be noted that the failure shown in Fig. G1 occurred at a small fillet while that shown in Fig. G2 occurred also in a region of high stress concentration located at the base of one of the splines where a sharp reentrant angle is contiguous to a fillet. Several instances of failures of this part occurred in different engines and the locations varied showing that the failures were not confined to any special point in the member.

Case H.—Airplane streamline wires. There are of course many failures of airplane streamline wires. Almost invariably these failures are of the torsional variety having a 45° angle of failure. Fig. H shows an example of failures obtained in an alternating torsion machine. These failures are quite typical of those found in actual service. Although as installed in a plane the wire is directly stressed in tension, the imposition of vibratory forces from flight conditions and from engine vibration produces complex stresses which apparently introduce torsional effects. This causes failures which are similar to those observed in the laboratory under pure torsion testing.

These instances illustrate the wide field in which fatigue and corrosion fatigue phenomena operate. The obvious preventives of such types of failure are three. First comes careful design using the present knowledge of the mechanism of fatigue and corrosion fatigue and the data and information available on the fatigue properties of the metals and alloys to be used. Second comes manufacture in accordance with best standards and processes. And last, but also of great importance, there should be required painstaking inspection during manufacture and erection, and periodically thereafter during the operating life of the machine or structure.

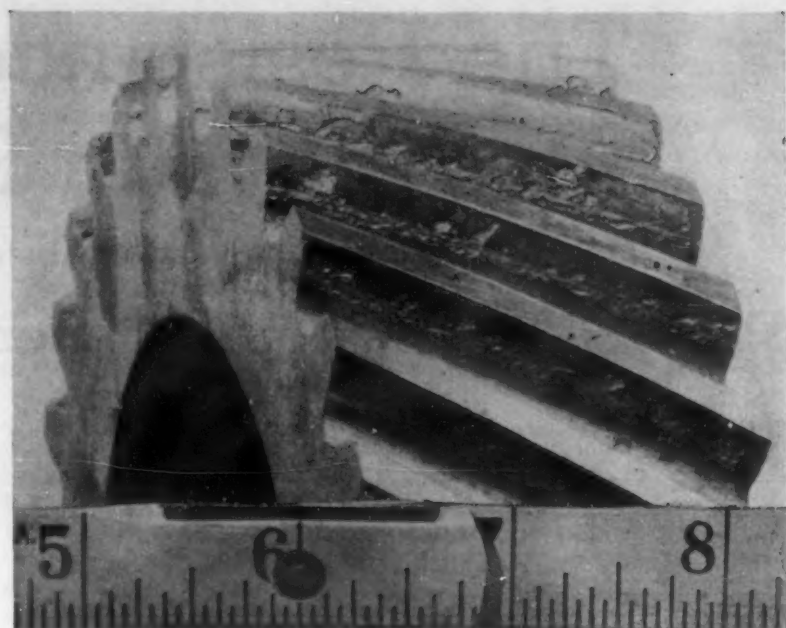


Fig. F. Fatigue failure of reduction gear pinion from excessive compressive stresses.

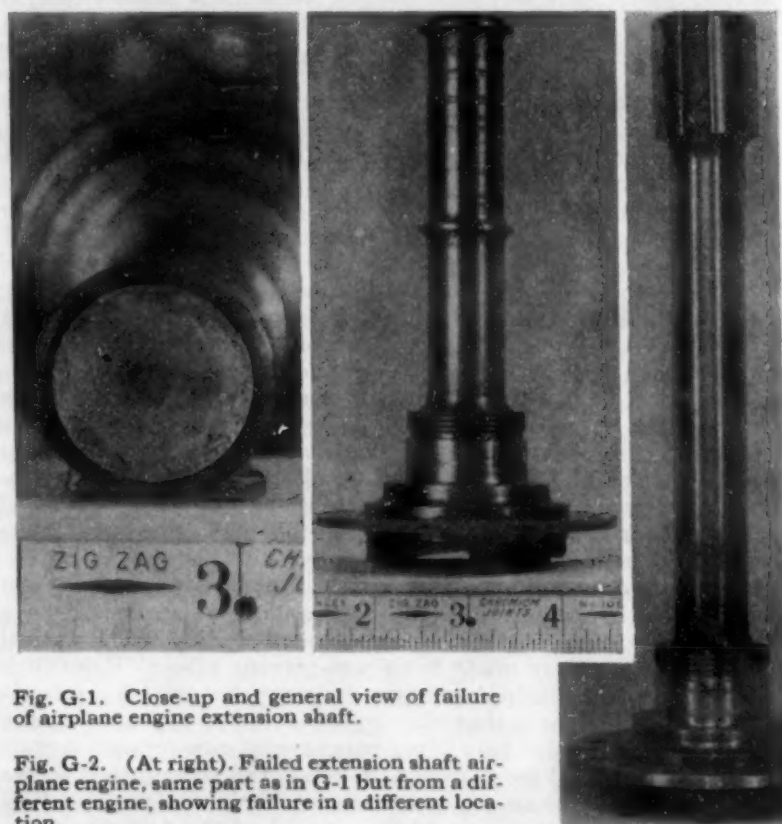


Fig. G-1. Close-up and general view of failure of airplane engine extension shaft.

Fig. G-2. (At right). Failed extension shaft airplane engine, same part as in G-1 but from a different engine, showing failure in a different location.

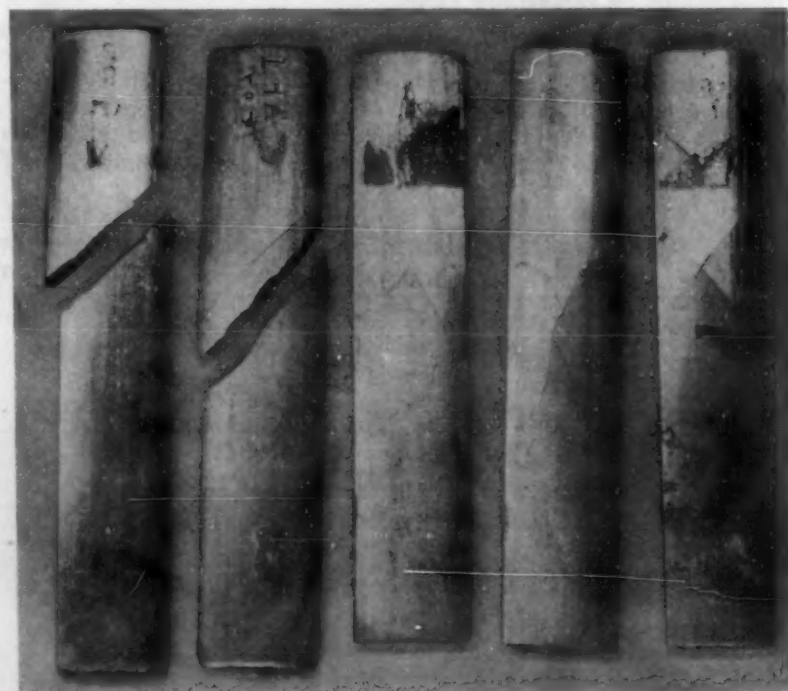


Fig. H. Failures of airplane streamline wires tested in alternating torsion endurance testing machine.

The Resistance of Copper and Its Alloys to Repeated Stress

A Correlated Abstract by H. W. Gillett*

Part I. Tough-Pitch Copper, Annealed and Cold-Worked

ENGINEERING design is paying more and more attention to the problem of failure of metals and alloys under repeated stress. The endurance properties of metallic materials of construction are very much in the lime-light. On account of high endurance limits, say up to 110,000 lbs./in.² or over, that may be obtained with heat-treated steels, much attention has been paid to the endurance properties of carbon and alloy steel, and they are used for most engineering purposes where fatigue-resistance is required.

Under combined corrosion and repeated stress, however, the ordinary steels fall from their high estate. McAdam¹ has cited a case where the corrosion-fatigue limit of a silicon steel in Severn River water is only 12,000 lbs./in.² although the endurance limit in air is 108,000 lbs./in.² Inglis and Lake² show that a mild steel under more severe corrosion in sea water really has no fatigue limit, but may be expected ultimately to fail no matter how small the repeated stress. On account of their corrosion-resistance, the copper alloys are useful to combat corrosion-fatigue.

The copper-base alloys have had relatively little attention from the fatigue point of view, probably because their applications as a rule do not involve the application of repeated stress. Some important uses do, however, involve it. Conductor cables swaying in the wind, propellers and propeller shafts, turbine blades, phosphor bronze cables for the steering gear of motor boats, copper bonds for electric railway rails, and, to a lesser degree, bearings, are subjected to repeated stress. Fourdrinier wires, used in the paper industry, may fail as a result of combined repeated stress, wear and corrosion. Copper or brass tubing for the gasoline or oil lines of aircraft, automobiles, etc. may also fail by fatigue. Piping in mechanical refrigerators has to withstand corrosion and vibration. Some control devices, notably the sylphon type of bellows, must undergo many repetitions of stress. Copper roofs expand and contract with changes in temperature and consequently have stress variations. Certain springs and switches for electrical contact purposes are made from non-ferrous alloys. Even such material as free-machining brass rod may be used for miscellaneous machine parts that may be subjected to repeated stress.

Moore and Jasper³ say, "One reason why non-ferrous metals have not been used more for parts subjected to repeated stress has been on account of ignorance of their fatigue-resisting properties. If engineers could be assured of the fatigue-resisting properties of a non-ferrous material, its range of usefulness would be materially extended."

Work on endurance of copper-base alloys has so far been, for the most part, incidental to the general study of fatigue and corrosion-fatigue phenomena, or to that of methods of endurance testing.

The alloys have had relatively little systematic attention from the point of their suitability as engineering materials of

construction for uses involving repeated stress. Such information is really more urgently needed for an intelligent choice among non-ferrous alloys than it is in the case of steels, because non-ferrous alloys as a class do not show even that approximate degree of correlation of endurance limit with static strength that holds in all but the very hardest steels. Increases in static strength obtained by alloying, coldworking, or heat treatment of a non-ferrous alloy may or may not be accompanied by corresponding improvement in resistance to fatigue; it is necessary to examine each particular case to find out.

The field has been fairly well surveyed in respect to some non-ferrous alloys. The endurance properties of nickel and Monel metal are quite well known. Those of aluminum and its alloys, and to a lesser extent, of magnesium and its alloys, have been quite thoroughly studied because of the importance of repeated stress in aircraft service. As a result of this intensive study, new aluminum alloys have been devised that are especially suitable to resist fatigue. Similar possibilities doubtless exist in the copper-base alloys, but have not yet been realized.

A good deal of information is available on endurance of copper base alloys, but it is so scattered that a summary should be useful.

The books on Fatigue of Metals, by Moore⁴ and by Gough,⁵ list hardly more than a dozen sets of data on copper-base alloys. One of McAdam's papers¹ collects some of the data from numerous other papers by himself, and Kommers⁶ has reported a systematic study on a few brasses of various compositions, but there has been no attempt to collect all available data, and to examine it for agreements and disagreements so as to bring out what is really known and what remains to be determined.

The bulk of the information is in published reports, but, in addition, new data or other information, and helpful comments and suggestions have been received during the working-up of this compilation from Dr. L. J. Briggs, Bureau of Standards; D. K. Crampton, Chase Brass & Copper Company; T. S. Fuller, General Electric Company; Prof. W. E. Harvey, Lehigh University; T. F. Hengstenberg, Westinghouse Electric & Manufacturing Company; Prof. J. B. Kommers, University of Wisconsin; J. B. Johnson, Wright Field; Dr. D. J. McAdam, Jr., Bureau of Standards; R. R. Moore, Consulting Engineer; Prof. G. Sachs, Metallgesellschaft; W. G. Schneider, Copper & Brass Research Association; J. R. Townsend, Bell Telephone Laboratories, G. S. von Heydekampf, Baldwin Southwark Corporation, and W. R. Webster, Bridgeport Brass Company.

The present review is confined to alloys containing not less than 50% copper. This first section deals primarily with tough-pitch copper. Later installments will deal with copper alloys.

In tabulating data, tensile strength and endurance limits are rounded off to the nearest 500 lbs./in.², and elongation and

TABLE 1. ANNEALED, WROUGHT ELECTROLYTIC TOUGH-PITCH COPPER ROD, TESTED IN ROTARY BENDING AT NORMAL FREQUENCIES

No.	Worker	Mark	Composition	Anneal	Tensile Strength lbs./in. ²	Elong. %	Red. Area %	Charpy Impact ft.-lbs.	Brinell	ENDURANCE				
										Stress, lbs./in. ² for		Endurance Limit		
										1 Million	10 Million	20 Million	lbs./in. ²	No. Million Cycles
1	Gough ⁵	ATV	99.96% Cu	*F. 1290	Time 1/2 hr.	29,000	60						12,500	(30)
2	Gough ⁵	BIF3	99.96% Cu, traces Fe, Ni, Sn	Annealed	32,500	59	74						11,500	(30)
3	Moore ³	102	99.98% Cu	1290 ?	32,500	56 1/2	72		47	14,000	11,000		10,000	(500)
4	McAdam ⁷	EE12	Not stated	1200 1 hr.	31,000	74	53	29 1/2		13,000			10,000	(100)
5	McAdam ⁸	DI12	Not stated	1200 1 hr.	32,000	57 1/2	72						10,500	?
6	McAdam ⁹	BU	Not stated	Hot Rolled	31,500	58 1/2	72						12,500	?
7	Ludwik ¹⁰		Not stated	Annealed	32,000	51	69 1/2		48		13,000			
8	Irwin ¹¹		99.92% Cu, .01% each Fe, Pb, Sn	1110 ?	31,000	60	69		47		10,500		10,000	(100)

*Battelle Memorial Institute.

reduction of area to the nearest $\frac{1}{2}\%$. Other data, as Brinell and impact, are similarly rounded off. The endurance ratio is included in the tables for those cases where the endurance tests have been run to 20 million cycles or more. In other cases, it is enclosed in brackets, a question mark added to show that the number of cycles was either not stated, or less than 20 million or in case of high-stress, low cycle tests, omitted entirely.

Many data on static properties are given in the original sources, especially in McAdam's work, which have not been incorporated in the tables. Proportional limit values are given by some other authors, but since the criterion is so variable, the values have not been included.

Since the life at stresses above the endurance limit is of interest, the stresses for lives of 1, 10 and 20 million cycles have been tabulated when the workers show complete curves.

ANNEALED TOUGH-PITCH COPPER. Table 1 shows that tough pitch annealed wrought copper has, in long-time endurance tests, an endurance limit for 100 million or more cycles of about 10,000 lbs./in.² or slightly more, as determined on rod by rotary bending tests.

Axial Loading. Irwin¹¹ has given axial loading tests, carried to 300 million cycles, for the same lot of annealed copper for which rotary beam data are given as No. 8, Table 1. These gave, for 1 million cycles, 12,500; for 10 million, 11,500; and for 300 million, 11,000 lbs./in.²

Sheet Data Lacking. All the data of Table 1 are for rod. No data were found on annealed copper sheet, outside of some data of doubtful validity which will be referred to later under the general discussion of the effect of speed of testing.

Tubing. The Army Air Service, at Wright Field, made rotary beam endurance tests on 99.96% Cu annealed copper tubing $\frac{3}{8}$ " outside diameter, 0.05" wall thickness. This had 34,000 lbs./in.² tensile strength, 56% elongation. When the endurance specimen was carefully polished inside and out, it showed at 1 million cycles 16,500; at 10 million, 13,000; and at 100 million, 12,000 lbs./in.² Tubing with ordinary commercial finish showed but 11,000 lbs./in.² at 20 million cycles. Some of this data is cited by Templin¹² and by Worthington.¹³ Aitchison and Barclay¹⁴ state that the British Air Ministry made fatigue tests of tubing in soft copper (9,000 lbs./in.²), dead soft copper (7,500), soft arsenical copper (10,000), dead soft nickel copper (indeterminate, probably 8,500-9,500). The footnote reference to the original is incomplete so it could not be consulted for explanation of these low figures, which might be due to scratches on the specimens, since copper is injured by the presence of notches.

Notch Effect. McAdam⁷ gives the endurance limit (number of cycles not stated) as only 3,000 lbs./in.² for bars carrying a notch of 0.0055 in. radius. The copper was annealed 2 hours at 750°F. and showed 30,500 lbs./in.² tensile, 58% elongation, 75% reduction of area, 42 ft.-lbs. Charpy. Moore and Lyon¹⁵ tested the same copper (No. 3, Table 1) on which they obtained an unnotched endurance limit of 10,000 lbs./in.², using bars with a round groove of $\frac{5}{64}$ " radius and found that the endurance fell to 6,000 lbs./in.² at 100 million cycles. Ludwik¹⁰ tested

the same copper (No. 7, Table 1) that gave 13,000 lbs./in.² at 10 million cycles on specimens with a notch of 0.05 mm. radius, 0.2 mm. deep, and also with an enlarged portion meeting the breaking section with a 0.5 mm. radius fillet and obtained, at 10 million cycles, 10,000 and 8,000 lbs./in.² respectively.

Torsion. Ludwik also tested this material in repeated torsion, obtaining 7,000 lbs./in.² torsion endurance at 10 million cycles. Few data are on record for repeated torsion of annealed copper in tests carried far enough to show the endurance properties.

Yuasa¹⁶ and Kidani¹⁷ have made torsion tests at high stresses and low numbers of cycles. Stromeyer,¹⁸ in work done before the requirements of testing methods for accurate determination of endurance limits were known, reports data extrapolated from repeated torsion tests carried only to a few hundred thousand cycles, from calorimetric measurements. He tabulates the torsion fatigue limits of phosphor bronze as 17,500 lbs./in.², and of commercial copper as 12,500, with no data for static properties, and no information as to the number of cycles used. The work is of interest only from the historical point of view.

Corrosion Fatigue. McAdam⁷ and Ludwik¹⁰ have tested annealed copper for corrosion-fatigue. McAdam found the same endurance limit 10,000 lbs./in.², (100 million cycles) as in air, while Ludwik found, for 10 million cycles, a drop from 13,000 lbs./in.² in air to 11,500 lbs./in.² in sea water.

Cast Copper. Only one figure was found for cast copper. Bartels¹⁹ made endurance tests on cast copper, cooled in the sand, of 37,000 lbs./in.² tensile, 10% elongation, and reports 19,000 lbs./in.² endurance at 10 million cycles. After welding and hammering, or welding, hammering and torch annealing, the stress for 10 million cycles fell to 10,000 lbs./in.² The analysis of the cast copper is not given. There seems to be no good reason for so high a figure as Bartels cites for the cast material, and the figure needs checking. The endurance ratio 0.51 is high for a non-ferrous alloy.

COLD WORKED TOUGH-PITCH COPPER. Table 2 gives data on cold-worked tough-pitch copper. There are no tests on axial loading save those on impure copper by Memmler and Laute which are given in Table 3. Data on sheet and tubing are scarce. The Wright Field data on tubing in Table 2 are for $\frac{3}{8}$ " O. D. tubing, 0.05" wall, polished inside and out. The endurance of tubing with commercial finish was 18,000 lbs./in.² at 100 million cycles.

Döring²⁰ found that boring a rather large hole through a specimen cold-worked to 48,000 lbs./in.² decreased the endurance, for 2 million cycles, 30 to 45% calculated on the reduced section.

Memmler and Laute,²⁶ however, found that a groove 0.03 mm. deep and 0.05 mm. wide did not reduce the endurance of the copper (No. 1, Table 3) that was cold-worked to 49,500 lbs./in.² tensile. McAdam⁷ has given notched endurance test data for material No. 4, Table 2, using a notch of 0.0055" radius. The number of cycles is not stated, but the endurance dropped to 4,000 lbs./in.² from 16,500 lbs./in.² for the un-notched material. He has also made corrosion-fatigue tests on this same material in salt water, and carbonate water, and found no reduction at all in the endurance limit.

TABLE 2. COLD-WORKED TOUGH-PITCH COPPER

No.	Worker	Mark	Composition	Condition	STATIC					ENDURANCE					
					Tensile Strength lbs./in. ²	Elong. %	Red. Area %	Charpy Impact ft.-lbs.	Brinell	Stress, lbs./in. ² for			Endurance Limit		
										1 Million	10 Million	20 Million	lbs./in. ²	No. Million Cycles	Endurance Ratio
1	Moore ⁸	102	Not stated	Cold-drawn*	56,000	6½	52		104	22,000	16,000		10,000	(200)	.18
2	Moore ²⁰		Not stated	Cold-drawn*	48,500	15	54		96	22,000			15,000	(100)	.31
3	McAdam ⁸	DI	Not stated	Cold-rolled Low Anneal	52,000	12	52½			25,000			16,000	(?)	.31
4	McAdam ⁷	HF2-5	Not stated	(3 hrs. 250°F.)	46,500	14	56	18		22,000			16,500	(?)	.35
5	McAdam ⁹	CL	Not stated	Cold-drawn	40,500	27	67			20,000			13,000	(50)	.32
6	Wright Field ²¹	Tubing	99.91 Cu		63,500	4				29,500	24,000		22,000	(100)	.34
7	Swinning ²²		"Commercial crude electrolytic"		58,500								16,000		.27
8	Crampton ²³	Sheet #20 B&S	Not stated	8 numbers hard	55,500	3½						20,500**		(16)	(.37?)
			Cu Oxygen												
9	Gillett & Mack ²⁴		99.95	.036	Cold-drawn	37,500	30	73		21,000			18,500	(50)	.49
10	Gillett & Mack		99.94	.049	Cold-drawn	37,500	29	67½		21,000			17,500	(50)	.46
11	Lehr ²⁵				45,000	17½				18,500	12,500				

*From $\frac{3}{4}$ " to $\frac{1}{4}$ " in one operation. **16 million cycles only.

Torsion. Moore²⁰ determined the torsional endurance limit of material No. 2, Table 2, at 100 million cycles, obtaining 10,000 lbs./in.² and checked this by finding that the endurance in repeated torsional loading from zero to a maximum (not reversed torsion as in the usual test) was 19,000 lbs./in.², while McAdam⁹ got but 4,000 lbs./in.² (number of cycles not stated) for material No. 5, Table 2, and on another sample of 40,500 lbs./in.² tensile, 24½% elongation, 66% reduction, he got but 6,000 lbs./in.² at 50 million cycles for the torsional endurance.

Becker and Föppel²⁵ using copper of but 99.3% Cu, static properties not stated, found 11,500 lbs./in.² torsional endurance at 10 million cycles.

Effect of Cold-Working. Considering Table 1, Table 2 and Tables 3a and 3b, we see that, in general, cold-working raises the tensile strength and the endurance limit.

Schule and Brunner²⁸ report the following early tests on repeated bending of 8 mm. copper wire:

	Tensile Strength lbs./in. ²	Elong. %	Life, No. of Repeated Bends (Millions) at a Load of 100 kg. Over a 4 mm. Radius
Soft	35,000	38½	0.6
Half hard	44,000	5	2.3
Hard	59,000	3¼	4.8 not broken
Originally hard, tinned 1 minute at 300°C.	37,000	24	2.5

Tests by Schwinning²⁹ indicate that drawing to small sizes may increase both the tensile strength and the resistance to repeated bending. Rods 4.5 to 4.55 mm. diameter gave 57,000 lbs./in.² tensile and a stress for 2 million cycles of repeated bending of 18,000 lbs./in.², while those of 2.5 to 2.85 mm. diameter gave 59,000 and 19,500 lbs./in.² But Schwinning's figures are lower than those of most other workers for material of lower tensile strength.

Döring,³⁰ using copper cold-worked to 48,000 lbs./in.² tensile, 11% elongation, finds its stress for a life of 2 million cycles 18,000 lbs./in.², and upon further cold-working of the surface (effective on most materials under rotary bending since it strengthens the most highly stressed outer fiber), he only raises the endurance some 8%. Work on similar cold-working of threads cut on this copper (by rolling with a special die made to conform to the thread against the base of the threads) was ineffective, whereas it was effective on many other materials.

Pape³¹ found, in work on failure of swinging copper wires, under test producing failure in 20,000 to 150,000 cycles (high stresses), that working the surface apparently improved the life 15 to 18% on the average, but the results showed wide scatter. The initial properties of the copper are not stated.

Eden, Rose and Cunningham³² tested copper cold-worked to 56,000 lbs./in.² tensile, and found it would stand only 20,000 lbs./in.² at 1 million cycles.

Moore and Howard³³ state that the endurance limit of annealed or cold-drawn copper is the same, 10,000 lbs./in.², but this refers only to Moore's own two tests. Moore's copper, cold-drawn from ¾" to ½" in one operation may have been over-cold-worked and harmful internal stresses, or even incipient cracks, set up.

The cold-drawn materials in Table 2 that have been only moderately hardened, or that have had a stress-relieving anneal seem to be better than those that are severely cold-drawn. Yet mere hardening by cold-work is not necessarily detrimental since Crampton's sheet (No. 8, Table 2) is as hard as Moore's rod (No. 1, Table 2) and has better endurance, while the Wright Field tubing is the strongest material in the table and has the highest endurance limit. For any particular mode of cold-working there may be an optimum degree of reduction beyond which the endurance will fall off although the strength increases. The difference of over 100% in endurance limit of Moore's rod and the Wright Field tubing is of real importance.

It would appear that for service requiring resistance to repeated stress, cold-worked copper should be superior to annealed copper, but that copper too severely cold-worked either generally or locally, may be worse than annealed copper.

Further work on endurance of annealed and cold-drawn copper is in progress in the laboratories of the Ontario Research Foundation, which it is hoped will throw further light on the subject.

The aircraft industry uses annealed copper tubing for oil and gas lines, and requires that they be periodically re-annealed to wipe out the "hardening effect of vibration." Worthington¹³ says this requirement is an erroneous one. It seems hardly possible that the effect of vibration would put the material into a state less resistant to repeated stress, since cold-working or under-stressing improves the endurance limit, and raises the "stress for 1 million cycles" also. Unless the real object is to remove excessive local cold-work induced by bending a fuel line sharply to get the tube out of the way of the workman in the course of maintenance work, which deformation might correspond to over-drawing, the requirement is not in line with laboratory tests. If practical experience were such that the requirement is actually a sound one, doubt would be thrown on the validity of conclusions drawn from endurance test data on rod stock.

The truth of the matter probably lies in the local over-working of the tubing by severe bending, since the indications are that severe over-working produces internal cracks or damage that will form nuclei for fatigue failures thereafter.

The provision for re-annealing at intervals would then be predicated on the assumption that damage done by over-working is repaired by annealing, a matter on which positive experimental evidence would be desirable.

One railway has raised a similar question. It is its practice to anneal the copper pipe and tubing used for locomotive oil and steam lines every 90 days, on the theory that the vibration in service hardens and embrittles the copper. The railway shop claims that dead soft copper will be hard and stiff when stripped from the locomotive, and even believes that it will harden to some extent on lying around a few months in the shop. No trustworthy evidence to support either claim has been put forward and requests for samples showing the phenomena have not been complied with.

The matter is of some importance, since it is stated³⁴ that half the failures in naval aircraft during 1931, due to power plant accessories (as differentiated from the main reciprocating parts of the engine) were caused by failures of the fuel system.

It is quite probable that the annealed tubing is the better because it will stand more severe bending in installation or in overhauls than would hard-drawn material, and it is also probable that when tubing has been bent in overhauling it is benefited by annealing, but it seems very unlikely that service vibration in any properly installed fuel and oil "plumbing" system injures it and the periodic anneals are probably senseless unless actual deformation has occurred.

On account of sensitivity to the notch-effect, i.e., to any local stress-raiser, fittings which form a sharp shoulder where the tubing enters the fitting may produce such stress concentration as to cause failure in service. A tube that has been in such a badly-designed fitting might be locally damaged and perhaps benefited by annealing, but the proper remedy would be a re-design of the fitting.

Experimental study of the endurance of copper and copper-base alloys cold-worked to different degrees of reduction by different methods should give useful information in establishing how far cold-working may go before the endurance ceases to rise and begins to fall. This should be supplemented by low temperature stress relieving anneals and by regular annealing tests to show whether damage done by over-working can be remedied by annealing. In this connection the recent comments of Wragg³⁵ are of interest.

TABLE 3a. DATA OF MEMMLER AND LAUTE FOR ANNEALED AND COLD-DRAWN IMPURE (ARSENICAL) COPPER IN HIGH-SPEED, AXIAL LOADING TESTS. ACCURACY DOUBTFUL

No.	Worker	Mark	Composition	Anneal	STATIC			ENDURANCE								
					Tensile Strength lbs./in. ²	Elong. %	Red. Area %	Stress, lbs./in. ² for 20 Million Cycles	Endurance Ratio							
"Commercial Cu" (Arsenical) Axial loading 500 cycles/sec.																
			Cu	Ni	As	Sn	Sb	Bi	Fe	Pb						
1	Memmler & Laute ²⁶	1	99.59	.03	.28	.012	.026	.022	tr	tr	1110° F. ½ hr.	32,000	47	74	13,500	.42
2	Memmler & Laute ²⁶	2	99.52	.07	.23	.03	.038	.004	tr	tr	1110° F. ½ hr.	32,500	49	69	11,250	.35
3	Memmler & Laute ²⁶	1	See above								Cold-drawn	49,500	7	69	16,500	.33
4	Memmler & Laute ²⁶	2	See above								Cold-drawn	37,500	10	85	under 13,000	.34

Axial Loading and High Speed Testing. The data of Memmler and Laute²⁶ in Table 3 are on axial loading in the high speed (Schenck) magnetically driven machine running at 30,000 cycles per minute. It is not clearly known what the effect of axial loading instead of rotary beam or reversed bending is, or what the effect of speed may be. Lehr²⁶ found for material No. 11, Table 2, 12,500 lbs./in.² in rotary beam test at 3,000 cycles/min., and on axial loading at 30,000 cycles/min., he obtained 22,500 lbs./in.² It seems unlikely that axial loading played much part in this discrepancy. Although Fischer²⁷ and others calculate that for engineering design (using steel) a larger factor of safety is required when axial loading is concerned than when the repeated stresses are in bending, and although Irwin¹¹ found a slightly higher endurance limit for axial loading (Haigh machine) than for rotating beam, in view of the results of France²⁸ (who, however, studied steels only), this conclusion seems doubtful.

From the practical point of view, when material is used in what is ostensibly axial loading, a generous factor of safety should be applied to the endurance limit as determined in rotary bending, but this is doubtless due to the fact that true axial loading is seldom obtained in commercial operations, and the true stress is above the stress calculated on true axiality, rather than to any great difference in the properties of homogeneous metals under the two types of loading.

The true effect of speed on testing is likewise very doubtful. Early tests by Jenkin²⁹ with a magnetically driven machine, on annealed copper originally containing 99.97% Cu, 0.03% oxygen, gave:

Frequency—cycles per sec.	Endurance—lbs./in. ² at 10 Million Cycles
50	11,000—
505	11,000+
973	12,000
1987	12,500

In later tests, Jenkin and Lehmann,⁴⁰ using tiny sheet specimens, vibrated by an air stream, appear on the face of the returns to have shown higher endurance limits for high rates of stress alternation. We have already commented⁴¹ on this.

The data are given, for material said to have 33,000 lbs./in.² tensile, 38 Brinell, as follows:

Frequency—cycles per sec.	Endurance—lbs./in. ² for 10 Million Cycles
50	10,500 (ordinary endurance test)
660	11,000
3200	13,000
5900	12,500
8800	12,500

The specimens were so tiny that stress measurements were obviously very difficult to make. They also state that in torsion at 4,000 cycles/sec. a copper tube, static properties not stated, stood 60 million cycles at 8,000 lbs./in.²

Jenkin's copper, containing 0.03% oxygen, was annealed for 30 minutes at 600°C. in an atmosphere of hydrogen, and this practice was also followed in the later work of Jenkin and Lehmann. T. S. Fuller of the General Electric Research Laboratory finds that such treatment will crack such copper to a depth of 0.02 in. so that, he states, the wire used by Jenkin must have been embrittled to the extent of $\frac{2}{3}$ of its section and the strip used by Jenkin and Lehmann over its whole section. Obviously, any conclusions drawn from data obtained on such material are of little interest.

To bring out the effect of a hydrogen anneal on copper containing oxygen, we may cite data from Bamford,⁴² who made a comparison of low and high arsenic coppers under rotary bending which indicates the strengthening effect of arsenic, but only a couple of bars of each composition were tested and stresses used which caused fracture in from 1 to 9 million cycles. The data follow. All specimens were annealed 20 minutes at 1110°F.

No.	As	O	Ni	P	Fe	Si	Tensile lbs./in. ²	Elong. %	Red. Area %
1	.31	.014	—	.079	tr	—	35,500	67	83
2	.53	.013	—	.196	—	.004	35,500	66	81
3	.05	.011	1.52	.116	—	—	38,000	60	79
4	.05	.084	—	—	.026	.002	31,000	53	67
5	.453	.098	—	—	tr	—	33,000	65	67

Repeated Stress Tests

No.	Stress lbs./in. ²	Life, Millions
1	15,250	6.6
2	14,000	6.0
3	18,000	1.2
4	12,000	1.4
5	14,000	8.5

Similar tests were then made on the various specimens after annealing $\frac{1}{2}$ hour in hydrogen at 1650°F., with the following results:

No.	Tensile lbs./in. ²	Elong. %	Red. Area %	Repeated Stress Test Stress lbs./in. ²	Life, Millions
1	34,000	63	75	15,000	0.4
2	34,500	60	78	13,500	2.3
3	44,500	40	62	17,000	1.8
4	20,000	10	7	11,500	0.009
5	20,000	11	6	13,000	0.003

None of this data gives the endurance limits, but it indicates the deleterious effect of hydrogen upon oxygen-containing copper. If Jenkin's copper was originally tough-pitch and then hydrogen annealed, as appears from his articles, sound copper should have stood very much higher stresses under his conditions, and the results would be worthless for evaluation of speed effects. Even the data of Lehr are open to question, because stress measurements at such speeds of vibration are extremely difficult.

The Bureau of Standards has developed a high speed air-driven fatigue machine, in which the specimen is floated on an air current. The apparatus is illustrated in Fig. 1, Vol. 1, page 145 of the October, 1929, issue of METALS & ALLOYS. The Bureau has used it on a wide variety of aluminum alloys in the form of strips cut from sheet $\frac{1}{8}$ " thick. The data are quite consistent⁴³ with ordinary slow speed results, and, for these materials at least, the indications are that there is no marked speed effect. The method is not as yet applicable to materials of very high endurance limit or to the very soft alloys of low yield strength. The Bureau was good enough to try out the method on materials supplied by the Copper and Brass Research Association, i.e., on copper of 54,000 lbs./in.² tensile strength, $7\frac{1}{2}$ % elongation, to which the apparatus was found inapplicable, and to brass of 26 $\frac{2}{3}$ % Zn and phosphor bronze of 7 $\frac{2}{3}$ % Sn, 0.01% P, the former of 91,000 lbs./in.², and the latter of 115,000 lbs./in.² tensile strength. The stress-cycles curve for the brass was a smooth one; the samples tested at 25,000 to 19,000 lbs./in.² broke at 3.7 to 9.9 millions. Specimens run at 17,000 and 16,000 lbs./in.² were unbroken after 13.1 and 17.5 millions respectively. While the test was run merely to see if the method was applicable to these materials and was not carried far enough to give the endurance limit, the indications are that it would lie below 19,000 and above 16,000 lbs./in.², which is consistent with the data of Townsend and Grenall for tests on sheet at ordinary frequencies.

The tests on phosphor bronze were not carried far enough to tell much, the stresses and number of million cycles for failure being, 35,000, 2.7; 26,000, 6.2; 24,000, 4.0; and 21,000, 5.1. Of these, the 26,000 lbs./in.² figure is out of line, or else the material shows considerable scatter. The other three points fall on the same curve as the one for the hard rolled brass and would point toward a somewhat lower endurance limit than some other tests on hard phosphor bronze. In view of this evidence, little credence can be given to the other high speed tests in the literature.

Over-Stressing and Under-Stressing. Gough⁴⁴ found that by stressing annealed copper at 16,000 lbs./in.² for 350,000 cycles, thus damaging it by over-stressing, an accelerated "deflection" test method showed the same indicated endurance limit (12,500 lbs./in.² as it did on virgin material, but the over-stressed speci-

TABLE 3b. DATA OF HANSON & FORD FOR ANNEALED & COLD-WORKED BISMUTH BEARING COPPER

No.	Worker	Mark	Composition	Anneal	STATIC			ENDURANCE		
					Tensile Strength lbs./in. ²	Elong. %	Red. Area %	Impact Izod ft.-lbs.	Stress, lbs./in. ² for 20 Million Cycles	Endurance Ratio
			% O % Bi							
1	Hanson & Ford ²⁷	CB002	.015 .002	1290°F. $\frac{1}{2}$ hr.	32,000	66	67 $\frac{1}{2}$	45 not broken	13,500	.42
2	Hanson & Ford ²⁷	CB005	.016 .006	1290°F. $\frac{1}{2}$ hr.	33,000	62	71 $\frac{1}{2}$	45 not broken	13,500	.41
3	Hanson & Ford ²⁷	CB015	.011 .016	1290°F. $\frac{1}{2}$ hr.	33,500	59 $\frac{1}{2}$	73 $\frac{1}{2}$	45 not broken	13,500	.40
4	Hanson & Ford ²⁷	CB01	.015 .015	1290°F. $\frac{1}{2}$ hr.	33,000	64	72	47 not broken	15,000	.45
5	Hanson & Ford ²⁷	CB002	.015 .002	Cold-rolled from $\frac{3}{8}$ " to $\frac{1}{4}$ " diam.	52,500	17	62	41 broke	19,000	.36
6	Hanson & Ford ²⁷	CB005	.016 .006	$\frac{3}{8}$ " to $\frac{1}{4}$ " diam.	53,500	17	64 $\frac{1}{2}$	47 broke	19,500	.36
7	Hanson & Ford ²⁷	CB015	.011 .016	$\frac{3}{8}$ " to $\frac{1}{4}$ " diam.	51,000	17	60	42 broke	19,500	.38
8	Hanson & Ford ²⁷	CB01	.015 .015	$\frac{3}{8}$ " to $\frac{1}{4}$ " diam.	51,000	13	73	43 broke	19,000	.37

Hanson and Ford also give some endurance data on copper containing Bismuth, cold-rolled to 48,500-51,500 lbs./in.² tensile and 2 to 8% elongation, but all the endurance bars showed signs of flaws at the fracture.

men, loaded again, and only to 11,500 lbs./in.², broke in less than a million cycles. Copper is apparently not as markedly strengthened by under-stressing as is steel. Gillett and Mack,²⁴ however, showed slight strengthening on one specimen of cold-worked copper with 0.036% oxygen, stressed at 18,000 lbs./in.² for 20 million cycles, and marked strengthening on another of the same copper at 18,500 lbs./in.² for 50 million. After this, it withstood nearly 20 million at 21,000 lbs./in.², and on raising the stress to 26,500 lbs./in.², it stood a further 10 million before fracture.

That stressing at or about the endurance limit alters the static properties, at least, is shown by Czochralski and Henkel.⁴⁵ Annealed copper of 33,500 lbs./in.² tensile, run at 11,500 lbs./in.² for three million cycles of an endurance test, then removed and tested in tension, rose to 36,000 lbs./in.² tensile, and one cold-worked to 51,000 lbs./in.² tensile, given one million cycles at 22,500 lbs./in.², then tested in tension, rose to 55,500.

Memmler and Laute²⁶ stressed annealed material at 9,000 to 18,000 lbs./in.², in steps, in endurance test, and calculated that this treatment raised the endurance limit from 13,500 to 14,500 lbs./in.² and the tensile from its initial 32,000 to 36,000 lbs./in.², while the ductility decreased slightly. One cold-worked to 49,500 lbs./in.² tensile, stressed at 11,500 lbs./in.² for 20 million cycles rose from 16,500 to 17,000 lbs./in.² endurance limit, and to 53,000 lbs./in.² tensile, also with decrease in ductility. This was on the high-speed axial loading endurance machine, and on impure copper.

The stress strain diagram, for bars run at or below the endurance limit, and subjected to tensile test, straightens out, and the proportional limit rises. It is evident that repeated under-stressing behaves much like cold-working, though no measurable deformation occurs. How near the endurance limit the stress must be, and how many cycles are required is not yet very clear.

Summary. There are no reliable data on endurance of cast copper. Annealed copper rod has an endurance limit of about 10,000 lbs./in.². Annealed tubing may run slightly higher. There are no reliable data on annealed sheet.

Cold-worked copper may vary in endurance limit from 10,000 to 22,000 lbs./in.², the former figure probably referring to material that is over-cold-worked. No data are available to show clearly what degree of reduction by various cold-working processes will produce optimum endurance, or to what extent damage done by over-working may be repaired by annealing. The bulk of the data on record are on rod. Such data may not be directly applicable to sheet and tubing.

Most available data were obtained by rotary bending. Very little is available on reversed bending or on axial loading. There are data which indicate a speed effect, but they are of doubtful reliability. The endurance properties of copper, even in the annealed state, are adversely affected by the presence of notches, fillets of too small radii, or any sort of stress concentration. Copper is practically immune from corrosion-fatigue in fresh or salt water.

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Improved Method of Polishing Metallographic Specimens of Cast Iron

By J. R. VILELLA*

THE method of polishing described in this paper was first made public in December 1930, at a meeting of the New York Chapter of the American Society for Steel Treating, held at the Union Carbide and Carbon Research Laboratories. Since then there have been numerous requests for copies of that talk, and for this reason it appears desirable to present the subject in somewhat more detail at this time.

The preparation of cast iron specimens for microscopic examination is frequently regarded as a routine polishing operation involving no special technique and one easily executed by the procedure employed for other metals. The fact that cast iron is unique in containing a very soft and friable constituent is not always taken into consideration. As a result, specimens of cast iron prepared by ordinary polishing methods invariably show more "graphite" than they actually contain. This anomaly is due to the fact that in average metallographic specimens the so-called "graphite flakes" are no more than enlarged cavities from which the actual graphite has been gouged out. Photomicrographs showing this type of "graphite" have frequently appeared in text books and in publications illustrating the effect of alloying elements on the flake size. For these reasons it was considered that the development of a reliable polishing method capable of revealing this constituent in its true proportions should be valuable to those interested in the metallography of cast iron.

It was observed during the course of this investigation that the graphite flakes were only gouged out during the final polishing. Observe in Figs. 1 and 2 that every particle of graphite is intact as the specimen comes from the last 2 grinding papers. Notice in Fig. 3, which is precisely the same field as Figs. 1 and 2, that after finishing on the cloth lap, every particle of graphite has been gouged out, leaving cavities much larger than the original flakes.

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Once these cavities are formed, the abrasive action of the polishing powder on their borders causes them to grow until they attain an apparently critical size, beyond which the rate of growth on further polishing is hardly perceptible. This apparent growth limit is reached when the borders of the cavities have been abraded to such an angle that the light they reflect enters the microscope objective. When this stage is reached, the polished surface is no longer flat, but wavy, the "graphite flakes" occupying the bottoms of the undulations.

When an improperly polished sample is examined with a high power lens of short working distance, many light rays reflected by the rounded borders of the flakes are collected by the objective, thus minimizing the pitted condition of the surface. At low power, however, due to the greater working distance of the lens, these inclined rays fail to enter the objective, and consequently any graphite flake whose borders have been slightly rounded will appear pitted. The criterion for correct polishing is therefore examination at low power.

To illustrate these observations a series of photomicrographs was prepared in the following manner. A specimen was carefully polished by the method to be described, taking care to preserve intact every particle of graphite so that the photomicrograph would be a faithful representation of the true quantity and size of the graphite flakes. This is shown in Fig. 4. Note that the flakes are thin, have smooth edges and are gray in color. The sample was then given an additional polish of exactly 10 seconds on a broadcloth lap charged with levigated alumina, and photographed at exactly the same spot as Fig. 4. Note in Fig. 5 that every particle of graphite has been partly gouged out, leaving cavities wider than the original flakes, having ragged edges and black in color. It was then polished for 15 seconds more on the same cloth and examined. Considerable growth of the cavities was detected. After 30 seconds it was found that the contour of the cavities had hardly changed, in-

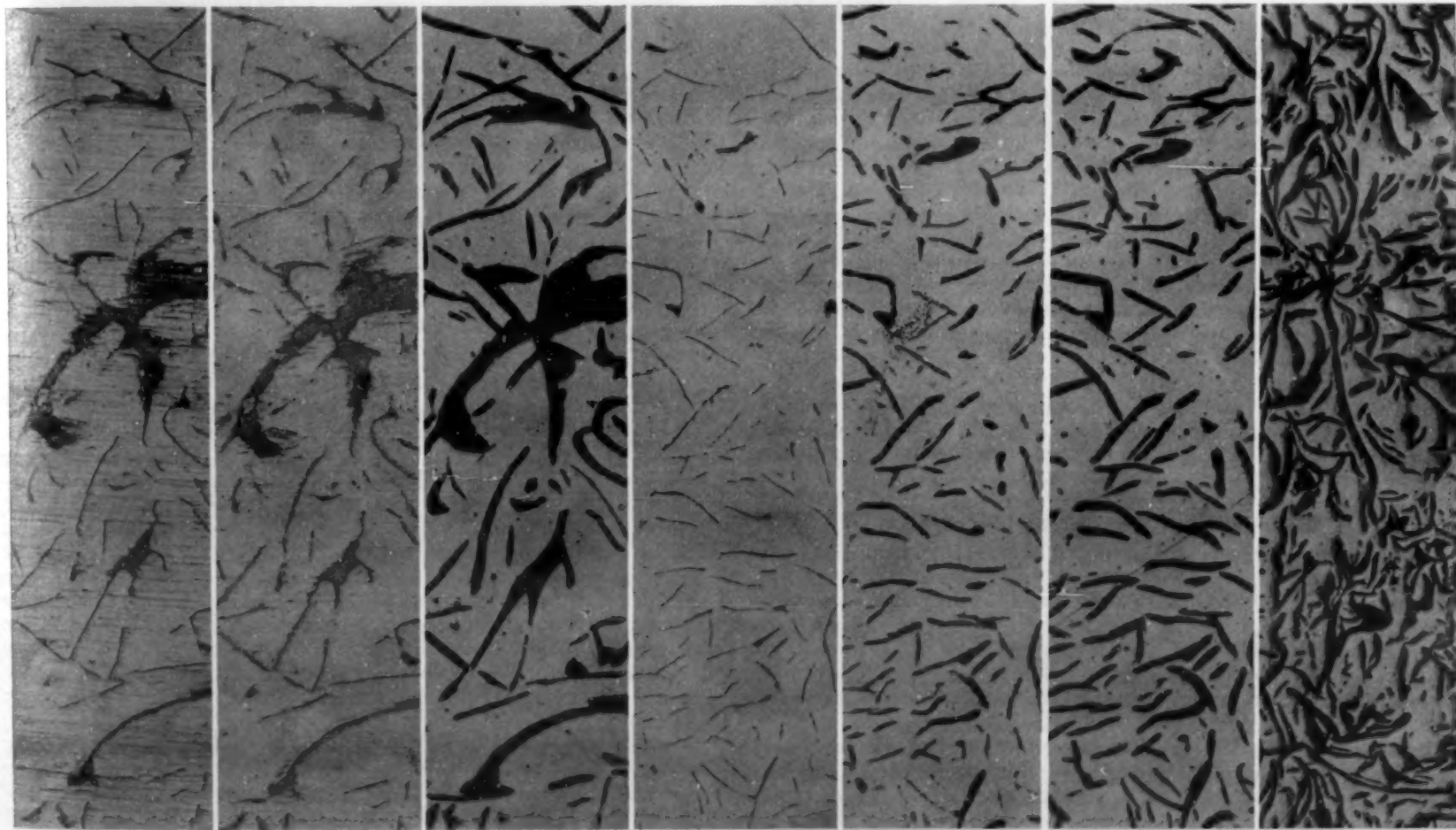


Fig. 1. Condition of surface after grinding on No. 000 paper. All flakes are intact. Mag. 100 X

Fig. 2. Same field as Fig. 1 after grinding on final paper. All flakes are intact. Mag. 100 X.

Fig. 3. Same field as Fig. 1 after finishing on broadcloth. All flakes dislodged, forming enlarged cavities. Mag. 100 X.

Fig. 4. Appearance of cast iron when properly polished. Mag. 100 X.

Fig. 5. Same field as Fig. 4 after polishing for 10 seconds on broadcloth. Mag. 100 X.

Fig. 6. Same field as Fig. 4 after 15 sec. 30 sec. and one min. additional polishing. Mag. 100 X.

Fig. 7. Same field as Fig. 4 photographed by conical illumination. Mag. 40 X.

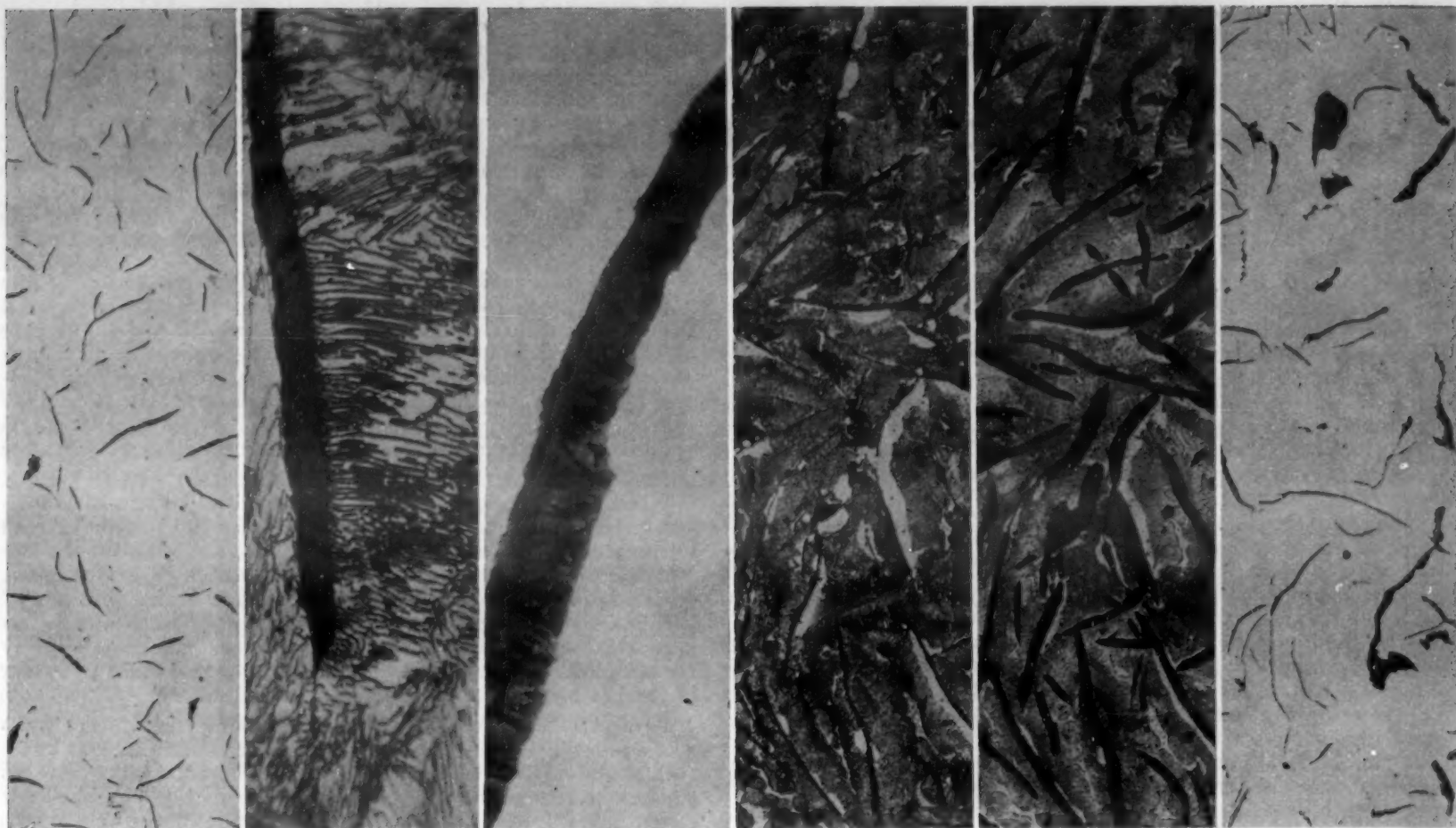


Fig. 8. Mag. 100 X.

Fig. 9. Etched with 2% nital Mag. 1500 X.

Fig. 10. A graphite flake. Mag. 3000 X.

Fig. 11. Properly polished. Notice areas of free ferrite. Mag. 100 X.

Fig. 12. Notice growth of cavities at the expense of free ferrite. Mag. 100 X.

Fig. 13. Showing dislodgment of shallow flakes lying parallel to the polished surface. Mag. 100 X.

dicating that the limit of rapid growth had been reached. An additional polish of one minute failed to appreciably increase the size of the cavities. The condition of the flakes at this stage is shown in Fig. 6. The polished surface is now made up of elevations and depressions, as may be seen in Fig. 7 which shows the same specimen photographed at lower power by conical illumination.

It follows from the preceding discussion that the solution of the problem of preserving the graphite flakes intact lies in finishing the sample on a suitable cloth. It has been shown that broadcloth dislodges the flakes. The same was found true of every soft fabric possessing a high pile or nap. To prevent dislodgment it was found necessary to employ a hard, pileless cloth. Linen and canvas were not satisfactory because they produced numerous scratches. By far the best results were obtained by using the dull side of a heavy, pure silk satin of good quality. This material does not distort the surface metal more than other fabrics; it does not polish in relief the hard constituents, nor in any way hinder the examination of etched specimens. Silk-finished surfaces that have been accidentally over-etched, stained or otherwise spoiled, can be repolished without pitting out the flakes. Examples of the results obtained by finishing on silk are shown in Figs. 8, 9 and 10. Note in the last pictures that graphite flakes when examined at high power exhibit a definite microstructure. This should not be confused with the roughness or granulation frequently exhibited by flakes that have been partly dislodged.

The effect produced on an etched surface by dislodging the flakes is illustrated in Figs. 11 and 12. Note in Fig. 11 the presence of considerable ferrite areas around the graphite flakes. In Fig. 12, which is the same field, repolished on broad cloth, these areas are not nearly as prominent. The formation of cavities has not only exaggerated the flake size but it has reduced the ferrite areas to a fraction of their actual size and number.

Inasmuch as the danger of gouging out the flakes exists only during wet polishing, the length of this operation should be reduced to a minimum. The use of intermediate wet abrasives like alundum or tripoli powder has not been found to yield the best results. Better graphite retention is obtained by finishing the grinding operation on a paper of such fine grit that its scratches can be readily effaced on the polishing cloth. To accomplish this purpose the following procedure has been found satisfactory:

Grind the sample at moderate speed on emery papers of in-

creasing fineness up to and including No. 000. It is important to press gently against this paper because any deep scratches produced at this stage will be difficult to efface during the next step. Then take the same sheet of No. 000 on which the sample was ground and glaze it by grinding on it a piece of graphite or soapstone. The use of No. 0000 paper is not advised because its grit is uneven. Use this glazed paper as the final grinding paper, again being careful to exert but gentle pressure. The type of scratches produced by the last two papers can be seen in Figs. 1 and 2. They should be fine enough to be totally eradicated on the silk lap in a short time. A sample one inch square should not take longer than 5 minutes.

The finishing powders must also be considered. Both levigated alumina and magnesia (heavy magnesium oxide) are satisfactory, but magnesia is preferable because it imparts a higher luster to the finished surface, produces fewer and finer scratches and shows less tendency to dig out the graphite flakes. It must not be suspended in water but applied in powder form to the wet cloth and worked in with the hand. Any gritty particles detected should be worked out. After using, the cloth should be carefully washed and rinsed with a dilute solution of hydrochloric acid to break down hard particles of magnesium carbonate which form on exposure to the air.

A certain amount of experience and skill is necessary before the best results can be obtained, particularly in the preparation and use of the last paper. Once these are gained, it will be found that the method is rapid, yields consistently good results and can be advantageously applied to other metals. It is strongly recommended for preparing steel samples that are to be examined for non-metallic inclusions. These are also very easily dislodged, but by the present method they can be preserved intact, irrespective of their size or composition, thus giving a true picture of the cleanliness of the metal. Only properly preserved inclusions can be identified by microscopic methods.

Attention must be called to the fact that even in carefully prepared specimens a number of cavities always occur. A few of these are due to porosity, but the vast majority are produced by dislodging flakes that lie nearly flat on the polished surface and are too shallow to stand much abrasion. (See Fig. 13.)

In conclusion, the author wishes to acknowledge valuable assistance received from D. Beregekoff in the preparation of the samples and in the form of helpful suggestions and criticisms.

READERS' COMMENTS

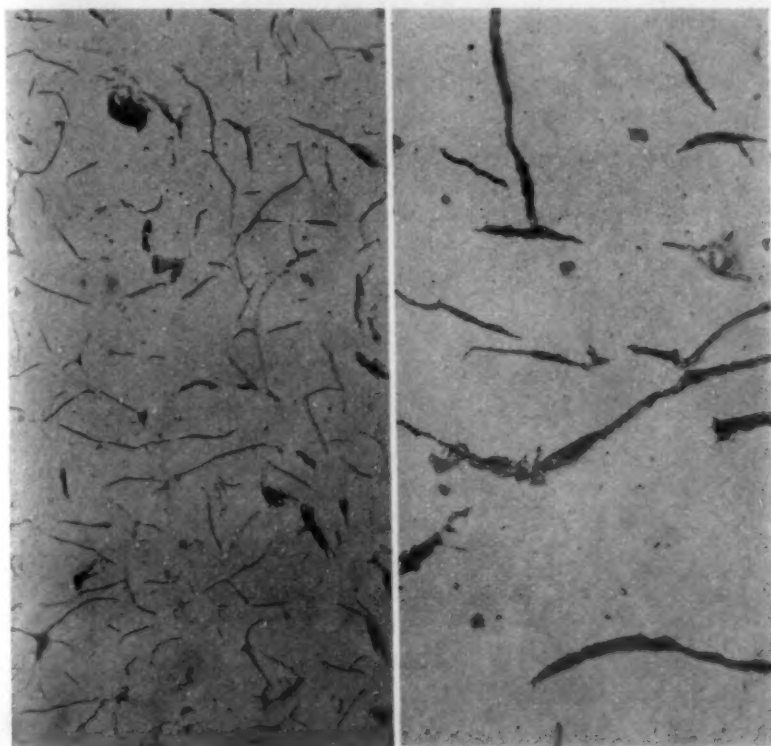
Editor, METALS & ALLOYS:

I have read the manuscript of Mr. Vilella's article with much interest. We can hardly have too many lessons in good polishing. It is well, too, to have alternate methods available, since the personal equation enters sufficiently into hand polishing to make different methods work best in the hands of different operators.

Vilella's method of going directly from the 000 grinding paper to the final polishing stage on a "silk lap" with magnesia powder has been very successful in his and Beregekoff's hands. The trick apparently lies in the gentle use of the grinding papers and in the final "glazing" of the 000 paper with graphite after it has been rubbed down considerably. The method probably requires practice. I have seen it tried by others with rather indifferent results, but there doubtless would have been an improvement as the operator gained experience.

The advantage of employing intermediate wet polishing stages is that less dependence is thus placed on the fine emery paper, the quality of which is often erratic. With a napless cotton cloth resembling linen in weave undue dislodgment can easily be avoided with alundum abrasives. This method probably takes no more time than Vilella's and perhaps less skill. Using alundum abrasives and thus depending less on the final grinding paper should be less costly because emery paper is rather expensive. In recommending a costly silk satin cloth for the final polish Vilella appears to be going to an unnecessary refinement since cheap velveteen cloth gives such fine results.

The attached micrographs are of a gray cast iron polished in the ordinary way through 000 emery paper then in turn with No. 320 alundum, No. 600 alundum, and levigated alumina on a napless cotton cloth, and finally with magnesia on velveteen cloth. The graphite flakes were virtually undisturbed and sharply outlined.



Mag. 100 X

Mag. 100 X

Micrographs of Gray Cast Iron polished with the use of intermediate abrasives on cotton cloth between emery paper and the final polish with Magnesia.

The above is not intended to gainsay the point Vilella has emphasized that polishing on the wet cloth wheels and especially with the intermediate abrasives is the source of pitting and dislodgment. This is why poor results are caused as often by too protracted as by too hasty polishing. It might be that for special purposes as where the structural features of the graphite flakes themselves were to be studied, Vilella's method would be preferable to the older one. In any event it represents a valuable contribution to the technique of metallographic polishing.

Aug. 10, 1932, Columbus, Ohio

SAMUEL EPSTEIN

FATIGUE OF METALS A Backward Glance

(Continued from Page 195)

corrosive action have been studied, and some progress has been made along this line.

The experimental study of the failure of metals under repeated stress brought out very sharply the ease with which a spreading crack could be started at a notch, a deep scratch, a screw thread, a small hole or some other "stress raiser."

The mathematical formulas of mechanics of materials have been developed mainly by civil engineers for use in designing structures, and they neglected thousands of high localized stresses, because under steady load no appreciable damage is done to the structure as a whole. However, under repeated loading each area of localized stress is a potential nucleus for a spreading fatigue crack.

Recent investigators with the microscope have confirmed the conclusions that fatigue failure is essentially failure by a spreading crack. The name "fatigue of metals" is not a good name, but it has become fixed even in technical language. It suggests a vivid analogy with failing muscular strength, and so far it has been impossible to supplant it by the more accurate term "progressive fracture." Later investigators found the amount of slipping which took place before a crack developed was very different for different metals. For some metals no evidence of slip was obtained before a crack appeared. Evidently metals differ widely in their ability to withstand occasional overload without starting a spreading crack.

The advent of the X-ray spectrograph and the work of Gough and others on single crystals has shown that this slipping action follows certain definite atomic planes within a crystalline grain, and that were it not for the interference between the variously oriented crystalline grains in a piece of metal slip would occur at ridiculously low stresses. But the question "just when and how does a fatigue crack start" is as yet unanswered.

The development of theories by students of fatigue of metals has clearly brought out the fact that whole mathematical theories of elasticity when applied to metals must be statistically true rather than absolutely true. It seems that the theory of elasticity is least accurate when used to determine stresses and strains over minute areas. So far, however, it has always been found to give results on the *safe* side, and the principle, abhorrent to the scientist, but helpful to the engineer, namely that an *untrue* theory which is known to be on the *safe* side may be useful, will doubtless keep the theories of elasticity in service for a long time to come.

The second boom period for the study of fatigue of metals seems to have reached its peak, and the study of "creep" of metals under steady load, a quite different form of failure, seems to be moving toward the spotlight. And the drama of research in metals goes on.



Norman W. Foy has been appointed Chicago District Sales Manager of Republic Steel Corporation. Mr. Foy became associated with the old Republic Iron and Steel Company as a salesman in 1919. He was subsequently manager of the company's Buffalo, Boston and Birmingham offices, being made Ass't Western Manager of Sales shortly after the formation of the present Republic Corporation. Mr. Foy will continue to make his headquarters at Republic's district sales offices in the McCormick Building, Chicago.

CURRENT METALLURGICAL ABSTRACTS

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